

TECHNICAL PROGRESS REVIEWS

REACTOR CORE MATERIALS

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NOVEMBER 1958

VOLUME 1

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TECHNICAL PROGRESS REVIEWS

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REACTOR CORE MATERIALS

a review of recent developments in
solid materials for reactor cores

prepared by
BATTELLE MEMORIAL INSTITUTE

NOVEMBER 1958

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foreword

In late 1957, the Division of Information Services of the U. S. Atomic Energy Commission launched a series of quarterly Technical Progress Reviews designed to make known the latest technological progress in the nuclear reactor field. With the publication of this issue, the series, *Reactor Core Materials*, completes its first year.

In writing these Reviews, the staff members of Battelle Memorial Institute have endeavored to select enough of the specific results and findings to permit an appraisal of their significance. References are provided at the end of each section for the benefit of those who wish to delve more deeply into particular areas. Readers are urged to consult these references for background information and for the original author's interpretation.

R. W. DAYTON

E. M. SIMONS

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REACTOR CORE MATERIALS

FUEL AND FERTILE MATERIALS

An aid to the researcher in fuel element technology may be the *Bibliography on Solid Fuel Elements*, recently published by the Sylvania-Corning Nuclear Corp., Bayside, N. Y. It contains 306 references and brief abstracts on fabrication and properties, most of which were published in 1956 and 1957.

Unalloyed Uranium

Mechanical-property determinations of beta heat-treated and irradiated uranium were performed at Hanford.¹ Room- and elevated-temperature tensile tests were performed on uranium exposed to burn-ups of up to 0.075 at. %.

although it did show a 45-deg shear failure; specimens tested at 600°C had square brittle-appearing fractures. It is suspected that differences in the mode of deformation between the high and low alpha range are responsible for the values obtained.

Du Pont³ studied the preferred orientation present in cold-rolled and recrystallized uranium plate. Cold working of uranium fuel elements after beta transformation is usually avoided since a preferred orientation may be produced which would result in dimensional changes during irradiation. However, the degree of cold work that uranium can undergo

Table I-1 ELEVATED TEMPERATURE PROPERTIES OF IRRADIATED URANIUM*

Exposure burn-up, at. %	Test temperature, °C	Ultimate strength, 1000 psi	Yield strength (0.2% offset), 1000 psi	Elongation in 1 in., %	Reduction of area, %
0.018	Room	78.0 av.	57.0 av.	0.75 av.	
0.031	Room	83.0 av.	73.0 av.	0.7 av.	
0.075	Room	83.0 av.	79.0 av.	0.5 av.	
0.018	300	46.0	40.0	16.0	35.0
0.031	300	54.6	50.4	2.23	
0.075	300	61.4	57.0	0.95	
0.018	450	24.5	19.4	12.8	
0.031	450				
0.075	450	26.0	21.5	0.51	
0.018	600	9.4	4.9	5.8	
0.018	600	9.9	7.2	5.7	
0.031	600	8.3	5.9	4.5	
0.075	600	5.8	4.2	1.4	

* Data from Hanford Atomic Products, Richland, Wash.¹

The results of these tests are shown in Table I-1. The most significant feature of these tests is the ductility exhibited by the low-exposure material. The sample tested at 300°C showed pronounced "necking" before fracture. The specimen tested at 450°C did not exhibit necking

before dimensional instability becomes excessive is not well established. In this investigation, beta-transformed uranium plates were cold-reduced 5 and 10 per cent to determine the amount of preferred orientation induced by cold working. When the anisotropy of thermal

expansion is used as a measure of preferred orientation, the expected linear growth of the plate, clad in aluminum, is estimated to be 1 per cent per 1000 Mwd/ton exposure for 5 per cent cold reduction and 2 per cent per 1000 Mwd/ton for 10 per cent cold reduction. Recrystallization following cold work had no significant effect on preferred orientation, but it did relieve intergranular stresses which affect the dimensional stability of the element. The cold work and recrystallization markedly reduced the grain size of the metal, an effect which might lessen the surface roughening of the plate during irradiation. A somewhat similar investigation at Chalk River⁴ was carried out on tubular NRX rods 1.36 in. in diameter. In this study, beta heat-treated uranium was cold-drawn to reductions of 0.70 to 5.8 per cent. Irradiation tests on this material showed that the growth rate as a function of cold work could be expressed

$$\text{Growth rate in } \mu\text{in.}/(\text{in.})(\text{Mwd/ton}) = 5.9 \times \\ (\text{per cent reduction in area}) - 2.4$$

This gives growth rates that are almost triple those estimated by Du Pont.

Swedish investigators⁵ have determined the diffusion coefficient of Xe^{133} in irradiated reactor-grade uranium in the temperature range 570 to 1000°C. The diffusion process can be described by means of an activation energy of 2.6 ev/atom (60 kcal/mole) to give a diffusion coefficient $D = 2.4 \exp(-59,600/RT) \text{ cm}^2/\text{sec}$.

British investigators have continued their study of the effects of heat and pressure on the swelling of irradiated uranium.⁶ Small pieces of natural uranium irradiated below 300°C to a maximum of 0.4 per cent fissioned atoms were heated to high temperatures under vacuum and various pressures. Heating in vacuum produced swelling of about 2 per cent at 575°C, 4 per cent at 810°C, and over 20 per cent at 1000°C. Repeated thermal cycling and reheating at high pressure produced swellings of 10 to 20 per cent at 810 to 835°C. Microscopic examination of the swollen metal revealed bubbles between 10^{-2} and 5×10^{-6} cm in diameter. Cracks were observed to be associated with nonmetallic inclusions and were particularly prevalent in thermally cycled specimens.

The following conclusions were reached from these experiments:

1. Irradiated uranium swells when heated to above 450°C.

2. Swelling can be reduced but not eliminated by the application of pressure.

3. After simple heating most of the gas is contained in bubbles with a radius of 0.2 μ .

4. The spacing and size of the bubbles produced when uranium is heated after irradiation are smaller than those of bubbles produced when it is heated during irradiation.

(M. S. Farkas)

Alpha-Uranium Alloys

Investigations by the British⁷ show that the uranium-vanadium eutectic temperature is lowered from 1040 to 740°C by nickel additions up to 8 wt.%. A chromium addition of 1/2 wt.% will lower the eutectic about 15°C, and molybdenum evidently elevates the melting point.

An investigation on the corrosion resistance of high uranium alloys is in progress at Battelle.^{8,9} The objective is to develop an alpha-uranium alloy that has a thermal-neutron-absorption cross section less than or equal to that of a 4 wt.% zirconium alloy and that is more corrosion resistant than uranium-2 wt.% zirconium in 300°C water. Seventeen arc-melted alloys were fabricated into specimens measuring 1 by 1/4 by approximately 1/16 in. The results of corrosion tests conducted on these alloys are shown in Table I-2. These tests indicate that for the conditions investigated:

1. Platinum, aluminum, niobium, and possibly ruthenium, when added to uranium-2 wt.% zirconium alloys in amounts as determined by cross-section considerations, improve the corrosion resistance more than an additional 2 wt.% zirconium does.

2. The corrosion life of uranium can be increased by a factor of 2 or 3, and the life of uranium-2 wt.% zirconium can be increased by a factor of 1.5 to 2 by ternary alloying. The degree of improvement in corrosion life varies with the heat-treatment.

Table I-2 CORROSION RESULTS OBTAINED ON URANIUM ALLOYS TESTED 15 MIN IN 300°C WATER*

Specimen	Nominal composition (balance uranium), wt. %	Weight loss, mg/(cm ²)(hr)†				
		Test 1	Test 2	Test 3	Test 4	
		1 hr 900°C, F.C.‡	3 min 800°C, W.Q.,‡ 6 hr 690°C, W.Q., 1 day 450°C, F.C.	1 hr 880°C, A.C.,‡ 1 day 450°C, F.C.	24 hr 880°C, A.C.	1 hr 900°C, F.C., 24 hr 450°C
1	Biscuit U		4040§	4760	4320	
2	2 Zr	1700	3068	> 3660¶	3328	3268
3	4 Zr	2080	3284	3160	> 3310¶	2960
4	5 Zr	2040	2888	2812	3140	2776
5**	2 Zr-0.04 V	2100	3184	4200		
6	2 Zr-0.33 Nb	1710	1496	2328	2692	2200
7	2 Zr-0.047 Ni	1690	3160	4040		> 2890¶
8	2 Zr-0.03 Ti	1760	2128	3640		
9	2 Zr-0.14 Mo	2250	3120	2992		
10	2 Zr-0.05 Pt	1360	1544	2440	1980	2528
11	2 Zr-0.07 Cr	1760	2616	3782		
12	2 Zr-0.015 V- 0.15 Si-0.01 Mo	2230	3128	Disintegrated		
13	0.015 V-0.15 Si- 0.01 Mo	2640	Disintegrated††	Disintegrated		
14	2 Zr-0.46 Al	1400	1916††	3120	2212	2360
15	1 Zr-0.5 Nb	1950	1568	2348	2712	
16**	2 Zr-0.15 Nb- 0.05 Mo	3110	2128	2600	2664	
17	2 Zr-0.16 Ru	1360	1300††	3216	2052	2520
	0.04 Cr-0.04 V- 0.1 Nb-0.2 Zr	2240	Disintegrated††	Disintegrated		

* Data from Battelle Memorial Institute, Columbus, Ohio.^{8,9}

† Based on original area.

‡ F.C. = Furnace cooled; W.Q. = Water quenched; A.C. = Air cooled.

§ As hot rolled.

¶ Disintegrated; rate based on original thickness.

** Chemical analysis shows these alloys exceeded the cross-section limit.

†† 1 hr 720°C, W.Q.; 1 day 450°C, F.C.

3. The corrosion rates of aged uranium-zirconium alloys are the same or slightly lower than those of the same alloys in the slowly cooled condition. However, the uranium-zirconium alloys with ternary additions of platinum, aluminum, niobium, or ruthenium show higher corrosion rates in the aged condition.

(M. S. Farkas)

Gamma-phase Uranium Alloys

Uranium-Niobium Alloys

The results of a study at Harwell¹⁰ on the uranium-niobium system have been published. A constitutional diagram and data leading to the diagram are presented.

Roof and Lombardo¹¹ have found that carbides observed in uranium-niobium-carbon alloys are complex carbides of uranium and niobium resulting from complete solution between uranium and niobium monocarbides.

In studies leading to the preparation of irradiation specimens at Harwell,¹² additions of 10 wt.% niobium were found to be required in order to stabilize the gamma phase in uranium-base alloys on quenching to room temperature. This value is higher than that found by U. S. investigators (about 7 wt.% niobium).

Battelle^{8,13,14} has been studying melting, fabrication, and homogenization procedures for uranium-niobium alloys. Attempts to reduce segregation in uranium-20, 40, and 80 wt.% niobium alloys by inert-electrode skull melting

have been made. Results were inconclusive due to the low pouring temperature employed. Consumable-electrode arc-melted material shows banding type segregation. Heat-treatments at 1200°F eliminate coring, but not banding. Similar results were obtained with heat-treatments of 4 hr at 1575°C and 1/2 hr at 1950°C for the 40 and 80 wt.% niobium alloys, respectively. The uranium-20 wt.% niobium alloy was rolled successfully at 650°C when jacketed in stainless steel. A 3/4-in.-diameter by 1/4-in.-long 20 wt.% niobium specimen, sealed in a stainless-steel tube, was upset forged and rolled at 700°C to 0.150 in. before the capsule cracked, after which the specimen was rolled out of a helium-atmosphere furnace at 700°C to 22 mils. A second stainless-steel-clad specimen was rolled to 90 mils at 700°C. A 40 wt.% niobium specimen, similarly encapsulated, could tolerate only a small reduction at 700°C. The uranium-80 wt.% niobium alloy, when jacketed with molybdenum, was rolled with partial success at 1000°C. The same alloy, sealed in a stainless-steel capsule, was cold press forged at a load of 300 tons.

600°C exposure, and only a small metallic fragment of the 80 wt.% alloy specimen remained. An 80 wt.% niobium specimen has shown a weight gain of 8 mg after 114 hr in 400°C air. Results of tests at 350°C are shown in Table I-4. Weight gains after 24 hr are 15 and 30 times greater at 400°C than at 350°C for the 20 and 40 wt.% niobium alloys, respectively.

Effects of zirconium additions on some properties and behavior of uranium-niobium alloys were investigated.^{8,13,14} The hardness of the 20 wt.% niobium alloy does not show a consistent trend with zirconium additions; an alloy containing 5 wt.% zirconium resists softening to as high as 900°C, whereas the alloy with 3 and 10 wt.% zirconium begins to soften appreciably at 500°C. After 1848 hr in 680°F water, the gamma-quenched uranium-20 wt.% niobium and -20 wt.% niobium-5 wt.% zirconium alloys exhibit corrosion rates of 0.03 and 0.02 mg/(cm²)(hr), respectively. The gamma₂-plus-alpha transformation temperature is lowered from 634°C in the binary uranium-10 wt.% niobium alloy to 594°C in the uranium-10 wt.% niobium-10 wt.% zirconium alloy.

Table I-3 ONE-DAY AIR OXIDATION OF URANIUM-NIOBIUM ALLOYS*

Nb content (balance U), wt. %	Specimen	Initial weight, g	Initial area, cm ²	Weight, g, after 24-hr exposure at indicated temperature		
				400°C	500°C	600°C
20	U-20-01	16.3090	10.0967	16.3740	17.0523	19.4273
40	U-40-01	17.7163	10.7213	17.7733	18.8183	22.8619
80	U-80-01	8.4745	7.4275	8.4769	8.5682	10.8939

* Data from Battelle Memorial Institute.¹⁴

Table I-4 LONGER TIME AIR OXIDATION OF URANIUM-NIOBIUM ALLOYS*

Niobium content (balance uranium), wt. %	Initial weight, g	Initial area, cm ²	Temperature, °C	Weight gain, g, after indicated exposure						
				24 hr	48 hr	72 hr	96 hr	120 hr	144 hr	168 hr
20	16.1170	10.063	350	0.0039	0.0063	0.0084		0.0128		0.0168
40	17.7611	10.736	350	0.0017	0.0045	0.0052		0.0048		0.0096
80	8.2852	7.3011	400	0.0012	0.0039	0.0039	0.0059	0.0064	0.0077	

* Data from Battelle Memorial Institute.⁸

Air oxidation tests of the uranium-20, 40, and 80 wt.% niobium alloys were also performed. Specimens were heated successively at 400, 500, and 600°C for 24 hr. Results are given in Table I-3. The 20 and 40 wt.% niobium specimens were completely oxidized after the

Uranium-Molybdenum Alloys

The first low-temperature in-pile irradiation tests on uranium-9 wt.% molybdenum alloys have been completed by Bettis.¹⁵ These confirm the postulated role of fission spikes in

causing phase transformations in this alloy. Kinetics of transformation are dependent upon interlamellar spacing and fission rate of the material.

In loop corrosion tests of clad and defected uranium-molybdenum alloys,^{16a} it was found that contamination of the system was small as long as corrosion rates were low, but it became significant when splitting of the cladding accompanying fuel-element failure occurred.

Examination of zirconium-clad uranium-10 wt.% molybdenum coextruded fuel rods to determine their conformance to specifications is reported by APDA.¹⁷ Specifications on dimensions, straightness, surface finish, weight, surface flaws, rod ends, and bond were met. However, allowable cladding-thickness variations of ± 0.0005 in. on the 0.004-in. cladding were exceeded.

The effect of ternary additions on uranium-molybdenum-base alloys is being studied by Battelle.^{8,14} Transformation studies indicate that 1 wt.% niobium additions to uranium-10 and 12 wt.% molybdenum alloys improve gamma thermal stability and increase hardness at temperatures to 900°C, whereas 3 and 5 wt.% niobium additions decrease stability and hot hardness. A 2 wt.% ruthenium addition also increased gamma stability. Quaternary combinations of ruthenium and niobium additions lead to decreased stability compared with the ternary alloys. A 7.5 wt.% molybdenum-2 wt.% ruthenium alloy is reported to be corroding at a rate of 0.28 mg/(cm²)(hr) after 1848 hr in 680°F water.

Harwell reports¹² that the addition of 6 wt.% molybdenum to uranium stabilizes the gamma phase in uranium alloys on quenching.

Ternary Alloys

Battelle hot-hardness data¹³ show that increasing niobium and molybdenum additions to a uranium-40 wt.% zirconium alloy results in decreased as-quenched hardnesses due to increased gamma stability and increased high-temperature hardness by virtue of solid-solution hardening. Additions of 3 wt.% niobium to 30 and 40 wt.% zirconium alloys and additions of 3 wt.% molybdenum to the 30 wt.% zirconium alloy lower the temperature of gamma decomposition from 606 to about 584°C.

Battelle studies of uranium-zirconium-niobium alloys show that 3 and 5 wt.% niobium ad-

ditions to the uranium-23 wt.% zirconium alloy lower the temperature at which alpha begins to form in conjunction with gamma from 685 to 655 and 638°C, respectively. The temperature at which gamma decomposes to the intermediate uranium-zirconium epsilon phase is also lowered. Niobium is found to increase the thermal stability of the gamma phase in these alloys. Alloys of 23 wt.% zirconium-5 and 6 wt.% niobium are found to possess excellent corrosion resistance in 680°F water when gamma quenched and then annealed at 500°C for 24 hr. Precipitation of a discontinuous alpha-uranium phase and resultant alloy enrichment of the gamma phase is presumed to be responsible. Alloys lower in zirconium generally exhibit optimum corrosion resistance when either water quenched or air cooled. Annealing at 500°C produces sufficient alpha uranium to destroy corrosion resistance. Corrosion data are given in Table I-5.

(A. A. Bauer)

Dilute Uranium Alloys

Aluminum-Uranium Alloys

As a part of a study concerned with developing a fabricable aluminum-48 wt.% uranium alloy, Nuclear Metals¹⁸ prepared castings of both a binary aluminum-48 wt.% uranium alloy and a ternary aluminum-48 wt.% uranium-3 wt.% silicon alloy. X ray indicated the presence of microcracks. One section from the binary alloy was heat-treated for 24 hr at 600°C to form an entirely aluminum-UAl₃ structure. Another section of the binary which was not heat-treated contained traces of UAl₃. These sections, together with one cut from the ternary-alloy casting, were extruded into tubes and evaluated. Microporosity was visible in all but the heat-treated aluminum-48 wt.% uranium alloy. X ray indicated the presence of transverse porosity bands and large particles in the binary alloys which were not noticed in the ternary. Silicon does have a refining effect on the particle size, and it is suggested that its presence may reduce porosity. Continued work with centrifugal casting at Battelle,^{8,13,14} indicates that, with development of proper feeding techniques and casting conditions, good quality

Table I-5 CORROSION-TEST DATA FOR CERTAIN URANIUM-ZIRCONIUM-NIOBIUM ALLOYS*

Alloy comp. (balance U), wt. %	Heat-treatment†	Corrosion rate, mg/(cm ²)(hr)	Exp. time in 680°F water, hr
23 Zr-5 Nb	1 hr at 800°C, W.Q.;	0.10	4200
	24 hr at 500°C, W.Q.		
	24 hr at 800°C, W.Q.;	0.06	504
	24 hr at 500°C, W.Q.		
	24 hr at 800°C, W.Q.;	0.11	4200
	24 hr at 500°C, W.Q.		
	5 hr at 900°C, F.C.	0.16	4200
	24 hr at 900°C, W.Q.;	0.86	4200
	24 hr at 500°C, W.Q.		
	24 hr at 1000°C, W.Q.;	0.06	672
	24 hr at 500°C, W.Q.		
23 Zr-6 Nb	24 hr at 800°C, W.Q.;	0.06	504
	24 hr at 500°C, W.Q.		
	24 hr at 900°C, W.Q.;	0.04	504
	24 hr at 500°C, W.Q.		
	24 hr at 900°C, W.Q.;	0.17	4200
	24 hr at 500°C, W.Q.		
	24 hr at 1000°C, W.Q.;	0.11	672
	24 hr at 500°C, W.Q.		
15 Zr-5 Nb	24 hr at 1000°C, W.Q.;	0.03	504
	24 hr at 500°C, W.Q.		
	24 hr at 800°C, W.Q.;	0.06	504
	24 hr at 500°C, W.Q.		
10 Zr-5 Nb	24 hr at 1000°C, A.C.	0.05	504
	24 hr at 800°C, W.Q.	0.07	672
	24 hr at 900°C, W.Q.	0.10	504
10 Zr-6 Nb	24 hr at 1000°C, W.Q.	0.08	672
	24 hr at 900°C, W.Q.	0.10	504
	24 hr at 1000°C, W.Q.	0.10	672
	24 hr at 800°C, A.C.	0.06	672
	24 hr at 1000°C, A.C.	0.04	504

* Data from Battelle Memorial Institute.

† W.Q. = Water quenched; A.C. = Air cooled; F.C. = Furnace cooled.

hollow extrusion billets of aluminum-85 wt. % uranium can be cast in air. Pouring temperatures should be at least 2400°F, and a mold surface speed of about 700 fpm (700 rpm for the 4-in.-diameter castings) is at or near the optimum.

The suppression of the UAl_3 - UAl_4 reaction by the addition of silicon is being studied in an attempt to provide ternary alloying which will produce a more refined structure than that of the binary alloy. It has been suggested that the addition of silicon allows the formation¹³ of

USi_3 . This compound may form a stable solid solution with UAl_3 . The two compounds have the same crystal structure and a size factor of 6 per cent, both of which favor the formation of a stable solid solution. Elements such as germanium, lead, palladium, platinum, and tin have compounds of the USi_3 type and should react in the same way with UAl_3 . The theory of solid solution is being checked by studying specially prepared alloys containing these elements. Battelle¹⁹ has redetermined the liquidus temperatures for alloys containing from 13 to 50 wt. % uranium. These temperatures are higher than those reported originally and as shown in the following tabulation:

Aluminum-uranium alloys, wt. % uranium	Liquidus temperature, °C	
	Battelle	Gordon and Kauffman
17	755	710
17.5	780	730
24.5	954	850
30.9	1068	975
41.6	1190	1140
51.2	1265	1250

Savannah River Laboratories²⁰ have completed a detailed study of the bond zone occurring at a uranium-nickel-aluminum interface. Specimens were prepared by both step pressing and fluid pressure bonding. Two nickel-aluminum compounds and two uranium-nickel compounds were identified. These were Al_3Ni , Al_2Ni_3 , U_6Ni , and UNi_5 . Uranium dioxide was found either as a second phase in the UNi_5 layer or as a continuous layer between the U_6Ni and UNi_5 zones. These particles coalesced into a continuous layer with some heat-treatment and dissolved completely with additional heat-treatment. The presence of this UO_2 layer weakens the bond. Bond strengths of less than 10,000 psi are obtained with the continuous layer; on the other hand, if the UO_2 particles are dispersed in UNi_5 , the bond strengths are above 10,000 psi.

Zirconium-Uranium Alloys

Knolls²¹ has performed corrosion tests with a bare zirconium-3.8 wt. % uranium alloy in 200 to 400°F water to study the amount of uranium which might be released to the water. Weight-change data do not reveal loss of ura-

nium after 1500 and 1000 hr at 200 and 400°F; however, uranium analyses indicate a loss of uranium. At 600°F, uranium loss to the coolant was about 0.0027 mg/(dm²)(hr), and coupling the uranium to 2S aluminum in 200°F water accelerated the corrosion of the aluminum. Bettis²² reports the following conclusions as a result of corrosion studies performed on Zircaloy-base fuel alloys in high temperature water.

1. Corrosion of low uranium (3 to 8 wt.%) alloys with Zircaloy base is characterized by initial formation of a protective oxide which is often followed by a film breakdown and general spalling.

2. Corrosion resistance decreases with increased uranium.

3. Zircaloy-2-base alloys containing 3 to 8 wt.% uranium exhibit best corrosion resistance in the fabricated condition.

4. Zircaloy-3-base alloys exhibit best corrosion resistance after quenching from 950°C.

5. Addition of small amounts of boron greatly affects the sensitivity of corrosion resistance to prior thermal history. Zircaloy-2-base alloys with boron added have the best corrosion resistance after quenching from 950°C. Zircaloy-3-base alloys with boron added exhibit the best corrosion resistance after being furnace cooled from 550°C.

The transformation kinetics of alloys containing 8.85, 11.1, 14.3, and 20.7 wt.% uranium have been determined by Douglass et al.²³ Increasing uranium concentration retards the alpha-zirconium precipitation and accelerates the precipitation of epsilon. Direct quenching results in the formation of alpha prime-martensitic alpha-zirconium structure. The 20.7 wt.% uranium alloy is essentially 100 per cent epsilon prime.

The effect of postirradiation heating on swelling of zirconium-uranium alloys is being investigated in some detail. Bettis¹⁵ reports that Zircaloy-2-clad specimens of a zirconium-10 wt.% uranium alloy which had been irradiated to a burn-up of about 0.97 total at.% were heat-treated at both 700 and 760°C for 1/2 hr. The specimens heated at 700°C experienced no volume change; however, the specimens heated at 760°C exhibited appreciable swelling. Initial measurements indicated a thickness increase of 213 per cent. The following Zircaloy-2-clad specimens were subjected to transient temperature excursions at 760°C for 6 to 10 sec:

Composition (balance Zr), wt. %	Total burn-up, at. %
6 uranium	0.58
8 uranium	0.78
10 uranium	0.99
12 uranium	1.21

Moderate swelling occurred in every case. Progressively longer heat-treatments are in progress. (R. F. Dickerson)

Plutonium Alloys

Preparation of Plutonium Alloys

The co-reduction of plutonium alloys is being investigated at Chalk River.²⁴ Plutonium-aluminum alloys prepared by direct reduction of plutonium trifluoride and plutonium dioxide with aluminum have been discussed previously in this section. Several other plutonium alloys have also been prepared using this technique.

A plutonium-gallium alloy resulted from the complete reduction of 200 mg of plutonium trifluoride heated in an excess of gallium metal in a vacuum for 2 hr at 1150°C.

Plutonium-beryllium alloys were prepared by reducing both plutonium trifluoride and plutonium dioxide with beryllium in a vacuum. The plutonium trifluoride-beryllium reaction was complete after 25 min at 1125°C, whereas the plutonium dioxide reduction was complete after 30 min at 850°C.

A reaction between plutonium-trifluoride and silicon proceeded readily at 1300°C in a vacuum and resulted in the formation of a fluoride-free plutonium-silicon alloy in 15 min.

Thorium and plutonium tetrafluoride reacted exothermally at 650°C. The temperature rose to above 1100°C while 60 per cent of the initial 200 mg of plutonium tetrafluoride charge reacted to form a plutonium-thorium alloy. The remainder of the charge was reduced to plutonium trifluoride, which does not react with thorium.

A 2.5 wt.% plutonium-uranium alloy was prepared by heating a uranium-plutonium trifluoride mixture at 1200°C for 15 min. This represents a 20 per cent plutonium yield.

Plutonium trifluoride was reacted with liquid magnesium for 15 min at 800°C under a helium atmosphere to produce a 3 wt.% plutonium-magnesium alloy. However, plutonium recovery

was only 30 per cent, and magnesium distillation losses were high.

Plutonium-Zinc Alloys

In a study of the plutonium-zinc system at Los Alamos, the existence of three compounds, PuZn_2 , $\text{Pu}_2\text{Zn}_{17}$, and PuZn_x ($2 < x < 8.5$), has been revealed by metallographic analysis. PuZn_2 is face-centered cubic with $a = 7.747 \pm 0.001 \text{ \AA}$ and has a density of 10.56 g/cm^3 . $\text{Pu}_2\text{Zn}_{17}$ is hexagonal and isostructural with the corresponding uranium-zinc phase. PuZn_2 and PuZn_{17} melt incongruently, whereas PuZn_x melts congruently. There is no evidence of a eutectic in the system. A coefficient of expansion of $3.7 \pm 0.3 \times 10^{-6}$ per degree centigrade from room temperature to 480°C has been reported for the zinc-stabilized delta phase of a plutonium-10 at.% zinc alloy. The delta phase transforms to epsilon at 481°C and on continued heating begins to melt at 682°C . Solubility of zinc in the epsilon- and delta-plutonium phases appears to be between 5 and 10 at.%.

Plutonium-Aluminum Alloy

A plutonium-8 at.% aluminum rod was cold-swaged from a $\frac{1}{2}$ -in. diameter to a $\frac{1}{8}$ -in. diameter at Los Alamos. The procedure followed was as follows: swage through $\frac{15}{32}$ -, $\frac{7}{16}$ -, $\frac{13}{32}$ -, and $\frac{3}{8}$ -in. dies; anneal at 400°C for 1 hr; swage from $\frac{3}{8}$ to $\frac{1}{8}$ in. in eight steps using $\frac{1}{32}$ -in. increments in die sizes. The beginning length of the rod was 7 in., and the finished length was 112 in. (V. W. Storhok)

Thorium Alloys

Dwight et al.,²⁵ has described two compounds of thorium with metals of the noble-metal group. These compounds are ThOs_2 and ThIr_2 . It is quite likely that other compounds corresponding to compounds of thorium with copper, nickel, or gold exist in the thorium-osmium and thorium-iridium systems.

Pearlman²⁶ has reviewed the results of corrosion tests in sodium and organics of thorium and thorium-uranium alloys. At temperatures as high as 750°C , thorium and thorium-uranium alloys are inert in sodium and NaK, but they do corrode in these liquids by gettering the oxygen present in the sodium and NaK. Weight changes of specimens exposed to these fluids

appear to be a function of temperature and the oxygen content of the liquid metal. Tests of thorium in *o*-terphenyl suggest some sort of passivation process for the reaction of thorium with organics. At 371°C , an initial rapid rate of corrosion was observed, followed by a long period in which no additional weight loss occurred. An initial weight loss was also observed for thorium in *o*-terphenyl at 427°C , but a large initial weight gain was noted at 482°C . The latter effect suggests a breakdown of the organic.

Atomics International²⁷ reports that a thorium-9 wt.% uranium alloy shows a creep rate of 0.001 per cent per hour at a stress of 6000 psi at 600°C . A correlation was noted between the creep strengths and the rate of swelling of three different materials. This is in agreement with the theory that fission gases cause swelling in irradiated fuels by a viscous mechanism opposed by the creep strength of the fuel. It is reported that the thorium-7.6 wt.% U^{235} alloy to be used in a fuel loading of the Sodium Reactor Experiment (SRE) is expected to swell about 5 per cent in volume for an irradiation of 3500 Mwd/ton at a fuel-surface temperature of 650°C . Five specimens of thorium-10 wt.% uranium alloy showed volume increases of 2.4 to 4.8 per cent in irradiations at the Materials Testing Reactor (MTR) to burn-ups of 0.3 to 0.4 at.% (2500 to 3500 Mwd/ton) at 650°C .

Battelle¹⁴ irradiated six thorium-11 wt.% uranium samples to a nominal 1 at.% burn-up at 540°C . Of the six, three were affected only to the extent of being slightly bowed, but the other three were in various stages of deterioration. The causes of these anomalies are not indicated. (W. Chubb)

Dispersion Type Fuel Elements

At Atomics International,²⁸ a density times thickness gauge ($D \times T$) in conjunction with a gamma scintillation counter is being used to determine the uranium variation in cores of stainless-steel- UO_2 fuel elements. As a result of these studies the blending of sufficient uranium oxide-stainless steel powder to produce a large number of plates (batch blending) has been found to be superior to the blending of just enough powder to produce a single plate. Statistical analysis indicated that cores blended

in batches had greater uniformity of uranium from plate to plate, whereas the uniformity along one plate was practically the same for either blending method. With batch blending, the variation between plates and along one plate was 2.6 and 2.4 per cent, respectively, of the absolute uranium content. A recent report by Hausner²⁹ discusses the slip casting of stainless-steel-25 wt.% UO_2 bodies. In brief, the process consists in preparing a slurry or slip from a well-blended mixture of stainless-steel powder, uranium dioxide, ammonium alginate, and about 12 wt.% distilled water. The slip is cast into plaster-of-Paris molds and allowed to set overnight. The casting is then oven dried and sintered at 1350°C for 2 hr in dry hydrogen. The tensile strength of the sintered material is about 26,000 psi, with elongations of 8.3 per cent. Some of the advantages of the slip-casting process listed are: (1) the brittle ceramic particles do not break up during processing, (2) the ceramic particles are uniformly distributed and perfectly randomly oriented; and (3) metal-ceramic combinations can be produced in shapes of large dimensions.

Dispersions of UO_2 in stainless have been irradiated by Knolls at surface temperatures up to 700°C and at burn-ups up to about 50 at.% of the U^{235} . Results are reported on the performance of both flat-plate and pin-shaped fuel elements in which the core densities varied from 90 to 98 per cent of theoretical. These variations in densities resulted from a difference in fabrication processes, i.e., hot drawing, swaging, cold binder extrusion, etc. It is reported, in the case of pins, that the low-density material improved the radiation stability, even though the core structure was inferior to that of the high-density material. In addition to the radiation performance data, some calculations are shown on the theoretical expansion rates of UO_2 , stainless steel, and UO_2 -stainless-steel bodies as a result of fission-product buildup.

At Sylvania-Corning,³⁰ a series of powder mixtures containing 30 vol.% UN in molybdenum, niobium, Nichrome V, 40 at.% titanium-niobium alloy, and vanadium were vacuum hot-pressed at 1200°C. The resulting materials were reported to be quite porous. An alternative procedure, consisting in cold compacting at 50 tsi, vacuum sintering at 1200°C for 4 hr, then hot forging and annealing at 1200°C was investigated. The densities were quite high, ranging from 91 to 100 per cent of theoretical. After

metallographic examination, it was concluded that no reaction occurred between the UN and either molybdenum or niobium; a slight reaction occurred between UN and vanadium, whereas UN definitely reacted with both Nichrome V and the titanium-niobium alloy. Dispersions of 30 vol.% U_3Si_2 , which were prepared in the manner described above, reacted with each of the metallic matrices.

The UN-Nichrome V reaction is somewhat surprising in that Battelle³¹ reported no reaction between UN and elemental stainless-steel powders after extensive hot working at temperatures above 1200°C. (D. L. Keller)

Uranium Oxide Fuels

Additional data on UO_2 have become available on the technology and performance of this fuel in American and Canadian nuclear-power programs. Detailed reports on the design and results of experiments on UO_2 pellet fuel in the PWR program are presented in three excellent summary papers from Bettis.^{16a-c} Most of this latter work has been discussed in previous Reviews.

Fabrication of UO_2 -containing Ceramics

The Canadian Mines Branch, Department of Mines and Technical Surveys,³² investigated the effect of furnace atmosphere on the sintering of several UO_2 powders. They reported that, in a certain temperature range, the powders sintered to higher densities in argon, steam, nitrogen, wet argon, argon-oxygen, wet nitrogen, or nitrogen-oxygen than they did in hydrogen. However, the oxygen-to-uranium ratio of specimens fired in atmospheres other than hydrogen was high, which could lead to undesirable oxygen release during service. Specimens sintered at 1400 to 1500°C in a steam atmosphere, followed by cooling in hydrogen, were substantially stoichiometric and were equivalent in sintered density to specimens of the same materials sintered at 1700°C in hydrogen.

In connection with a study³³ of the effect of carbon on the sinterability, structure, and water-corrosion resistance of UO_2 ceramics, specimens were fabricated from UO_2 powder made from ammonium diuranate, or from starch-reduced anhydrous UO_3 . Pellets containing 0 to 10 wt.% graphite were pressed at 40,000 psi and sintered in a high-frequency

carbon-susceptor furnace or in a hydrogen-atmosphere alumina-tube furnace. The effect of graphite on sintered densities is shown in

Table I-6 EFFECT OF GRAPHITE ADDITION ON SINTERED DENSITY OF UO_2 COMPACTS FIRED IN A CARBON-SUSCEPTOR FURNACE*

Test	Carbon present, wt. %†	Type of UO_2	Sintered density, g/cm ³ ‡
1	0.27	Starch-reduced UO_2	9.96
2	0.5	From ammonium diuranate	10.30
3	1.0	From ammonium diuranate	10.01
4	2.5	From ammonium diuranate	9.17
5	5.0	From ammonium diuranate	7.86
6	10.0	From ammonium diuranate	6.36
			10.53

*Data from Canadian Bureau of Mines.³³

†This refers to carbon content of the compact prior to sintering.

‡Each observation is based on the mean of five compacts.

considerably by autoclaving at 300°C for 48 hr at a pressure of 1200 to 1250 psi. In view of the effect of graphite on sinterability and the deleterious effect of carbides on water-corrosion resistance, it was recommended that the carbon content of green UO_2 compacts be kept below 200 ppm.

Knolls³⁴ reported the results of sintering experiments on UO_2 - CeO_2 ceramics containing 21.3 to 82.1 wt. % UO_2 . Specimens were cold-pressed from mixtures of U_3O_8 or UO_2 and CeO_2 at 33,000 psi and sintered in air or hydrogen in the temperature range 1400 to 1950°C. All specimens sintered to low density (61 to 91 per cent of theoretical). Specimens made by mixing U_3O_8 and CeO_2 , then sintering in air, were of higher density and strength than those formed by mixing UO_2 and CeO_2 and sintering in hydrogen at the same temperature.

(H. D. Sheets)

Table I-7 EFFECT OF GRAPHITE ADDITIONS ON THE SINTERING BEHAVIOR OF UO_2 COMPACTS FIRED IN A CARBON-FREE ENVIRONMENT*†

Added graphite, wt. %	Green density, g/cm ³	Sintered density, g/cm ³	Oxygen-to-uranium ratio in sintered compacts	Graphitic carbon in sintered compacts, wt. %	Total carbon in sintered compacts, wt. %
Spectrographic SP-2 graphite additions					
1.0	5.13	9.51	2.006	<0.01	<0.01
0.5	5.29	9.86	2.012	0.01	0.21
0.25	5.27	9.84	2.009	0.02	0.02
0.10	5.30	10.39	2.008		0.01
0.05	5.37	10.31	2.006	<0.01	<0.01
	5.44	10.44			
Acheson Grade 38 graphite additions					
1.0	5.18	9.39	2.009	<0.01	<0.01
0.5	5.25	9.92	2.018	0.17	0.18
0.25	5.29	9.84	2.010	0.04	0.06
0.10	5.37	10.39	2.011	<0.01	<0.01
0.05	5.43	10.24	2.009	<0.01	<0.01
	5.44	10.44			

*Note: Uranium dioxide compacts with graphite additions fired to 1700°C for 1/4 hr in hydrogen. Density values are the mean of five compacts; analyses were made on a composite of three compacts.

†Data from Canadian Bureau of Mines.³³

Tables I-6 and I-7. Microscopic and X-ray examination of specimens sintered in the carbon-susceptor furnace showed the presence of UC and UC_2 in the sintered compacts. Specimens sintered in a hydrogen atmosphere had little carbon remaining after sintering. A specimen containing 0.3 wt. % carbon was damaged

Physical Properties of UO_2

Physical properties pertinent to UO_2 technology and UO_2 fuel performance are being investigated at various sites. A Canadian report reviews all thermal-conductivity data on UO_2 bodies and UO_2 powders in gaseous en-

environments.³⁵ Measurement techniques are surveyed and critically evaluated. Knolls³⁶ has investigated the thermal stability of Mallinckrodt UO_2 from room temperature to the melting temperature in hydrogen, helium, and argon. No effect of the type of gas on melting point was observed. A mean melting point of $2760 \pm 40^\circ\text{C}$ was determined in 20 experiments in these gases. No change in lattice constant was observed prior to melting. From room temperature to 2360°C , and probably to 2520°C , phase changes did not occur. It is concluded that Mallinckrodt UO_2 does not reduce to a lower oxide before or during melting in these gases.

At Battelle³⁷ the structure, composition, and chemical reactivity of uranium trioxide were studied to determine the effects of these variables on the reactivity of UO_2 made from various preparations of uranium trioxide. Depending on the preparation method, the thermal decomposition of uranyl hexahydrate yields either crystalline or amorphous UO_3 . The higher the nitrogen content of the UO_3 the lower is the reoxidation reactivity of UO_2 prepared from the trioxide. The nitrogen content of the UO_3 depends on the time and temperature of the denitration and pyrolysis method and the amorphous or crystalline nature of the UO_3 . When exposed to air, the change in the oxygen-to-uranium ratio of the UO_2 varies inversely with the nitrogen content of the trioxide.

There is some evidence that volatilization of uranium oxide may be responsible for local migration of UO_2 in pellet fuel at high temperature. Battelle is investigating the volatility of UO_2 in air, oxygen, and nitrogen in the temperature range 1100 to 1300°C . In these studies the gases are passed over UO_2 at temperature, and the amount of uranium oxide picked up by the gas is collected in a condenser. Vapor pressures of the volatile species, probably UO_3 , are calculated from the amount collected and the flow rate. It is assumed that the volatile species contains 1 atom of uranium per molecule. Typical results in oxygen and oxygen mixtures are given below in equation form. The partial pressure, p , is in millimeters, and the temperature, T , is in degrees centigrade.

At oxygen pressure 760 mm:

$$\log p = \frac{-17,456}{T} + 9.2019, \text{ from } 1146 \text{ to } 1487^\circ\text{C}$$

At oxygen pressure 150 mm, nitrogen pressure 610 mm:

$$\log p = \frac{-18,437}{T} + 9.7060, \text{ from } 1234 \text{ to } 1524^\circ\text{C}$$

At oxygen pressure 29 mm, argon pressure 731 mm:

$$\log p = \frac{-16,255}{T} + 8.1460, \text{ from } 1229 \text{ to } 1320^\circ\text{C}$$

$\text{UO}_2\text{-ThO}_2$

The irradiation performance of $\text{UO}_2\text{-ThO}_2$ pellet fuels studied by Argonne and discussed in previous Reviews has been reviewed recently in the ceramic literature by Hoenig et al.³⁸

(W. S. Diethorn)

Mechanism of Corrosion of Fuel Alloys

Workers at the Savannah River Laboratory have studied the mechanism of bond-line corrosion of aluminum-clad nickel-bonded uranium fuel elements in deionized water³⁹ at 95 to 98°C . It is believed that attack in metallurgically bonded samples containing an intentional pinhole occurred when hydrogen from the corrosion of uranium diffused along the bond layer and reacted with uranium to form uranium hydride. The hydride then reacted with water to form UO_2 and liberate more hydrogen. It was found that the hydrogen diffused along oxide particles in a UNi_3 layer in the bond zone. Attack occurred rapidly in UNi_3 layers in which the oxide particles were continuous and slowly in those layers in which the oxide particles were dispersed.

An investigation has been completed at Battelle on the corrosion of uranium in 100°C water and 200°C steam at atmospheric pressure.⁴⁰ Material balance in water and steam corrosion of uranium was studied by examination of the phase composition and valence state of the corrosion product and by hydrogen-evolution measurements. It was found that the composition of the newly formed corrosion product was $\text{UO}_{2.2}$, which increased to $\text{UO}_{2.4}$ after exposure to room-temperature air. The amount of hydrogen collected from the reactions indicated that the solid corrosion product should

have a composition of $\text{UO}_{1.9}$. Valence studies on the corrosion product indicated: (1) an oxygen-rich UO_2 type phase with the CaF_2 type structure, and (2) a phase which had a valence less than 4. X-ray diffraction analysis of separated residues from 100°C water tests revealed that the low-valence phase was metallic uranium particles incorporated in the corrosion product. It is believed that the presence of uncorroded metal accounts for the low-hydrogen assay.

No hydride was detected in the corrosion product nor in any of the corroded samples; however, its stability under the test conditions was verified. It is believed that a hydride layer previously observed in the corrosion product at temperatures above 100°C was due to an increased hydrogen pressure in the closed reaction vessels; in these experiments, hydrogen was allowed to escape from the reaction vessel and no pressure was built up.

Reactions with Carbon Dioxide

Personnel at Harwell have studied the reaction⁴¹ of CO_2 with uranium over the temperature range of 500 to 1000°C . They found that the reaction product on the uranium was UO_2 and that the reaction rates were linear with time. Reaction rates increased with temperature in the range 500 to 780°C , with a marked increase at or near the β - γ transformation temperature. Overheating of the uranium occurred at temperatures between 700 and 1000°C . Between 780 and 1000°C , the rate gradually decreased with increasing temperature. The decrease appeared to be a result of the formation of an adherent oxide layer which served as a barrier to further oxidation. At these temperatures, it is believed that the oxide scale is sufficiently plastic to reduce stresses by deformation rather than by cracking. In the range 440 to 725°C , the activation energy was calculated to be 27.9 kcal/mole, which is consistent with oxygen-ion diffusion being the rate-controlling mechanism.

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Beryllium

Beryllium Metal and Alloys

Personnel of Nuclear Metals, Inc.,¹ have made a study of the tensile properties of beryllium couplings, both mechanical (bolted or riveted) and brazed. Couplings examined were beryllium to beryllium, beryllium to 2S aluminum, and beryllium to type 316 stainless steel. The beryllium was in the form of a 0.060-in. sheet of basal-plane layered texture which exhibits two-dimensional ductility in the plane of the sheet. In the case of bolted or riveted lap joints, beryllium joined to itself undergoes various amounts of plastic deformation before fracture. When one component is stainless steel, most of the plastic deformation occurs in the stainless steel. Fracture, however, occurs through the beryllium. When one component is 2S aluminum, both plastic deformation and fracture are limited to the aluminum. In brazed couplings (when a 90 per cent aluminum-10 wt.% silicon brazing alloy is used), there appears to be no ductility whatever in the beryllium, regardless of whether it is joined to stainless steel, aluminum, or itself.

The coextrusion of beryllium and uranium to make beryllium-clad fuel elements² is also being studied. The technique is a multitemperature extrusion in which the billet components, consisting of a hollow beryllium cylinder and solid uranium cylinder, are heated in separate furnaces to the temperatures suitable for coextrusion. The components are assembled into the extrusion liner, upset to minimize thermal barriers at the beryllium-uranium interface, held a precise time to allow limited thermal flow, and then extruded. It was observed that the cracking tendency is less in the thicker beryllium wall. Some results of these experiments are presented in Table II-1. It can be seen that phase changes are likely to occur in the uranium during or after the extrusion process.

Mention has been made earlier of the research being conducted at Alloy Research

Corporation on the preparation of high-purity beryllium for intrinsic ductility studies. Although no high-purity beryllium has been produced as yet in this work, an interesting observation was made in conjunction with induction heating of beryllium for zone refining.³ When a 450-kc heater was used at full power, with a 1-in.-diameter two-turn coil, it was noted that the beryllium remained at a stable red heat for about 25 min, then suddenly got white hot. It

Table II-1 RESULTS OF SOME EXPERIMENTS ON THE COEXTRUSION OF BERYLLIUM AND URANIUM
(Data from Nuclear Metals¹)

Initial temperature of billets, °F	Uranium	Calculated temp. and extent of cracking at beryllium-uranium interface* for indicated conditions (see Note below)		
		1-in. rod, 10 sec after upsetting	3/4-in. rod, 30 sec after upsetting	3/4-in. rod, 100 sec after upsetting
2000†	900	1240 β (a)	1540 γ	1680 γ
2000‡	900	1210 α (b)	1515 γ	1660 γ
1800†	900	1090 α (c)	1360 β (d)	1440 γ
1600†	700	970 α (a)	1210 β (d)	1270 β (d)

Note: Extent of cracking in beryllium:

- (a) Moderate to severe.
- (b) Little.
- (c) Little to severe.
- (d) Moderate to little.

* α , β , or γ designates the allotrope of uranium existing at the interface temperature.

† 0.020-in. beryllium wall.

‡ 0.040-in. beryllium wall.

was suggested that the reason for this behavior might be due to the sluggishness of the suspected high-temperature transformation in beryllium, with the high-temperature phase coupling more strongly to the induction field. Further experiments are planned to study this behavior.

(A. J. Griest)

Beryllium Compounds

A new determination of the thermal conductivity of BeO was reported by Ditmars and Ginnings.⁴ A rod of hot-pressed BeO with density of 2.62 g/cm³ (equivalent porosity = 13.4 per cent) was used as the specimen for determinations at 30 to 748°C. The absolute method employed required no reference material but did necessitate careful adjustments to control temperature gradients in the test apparatus. Results of the measurements are summarized below.

Temp., °C	Thermal conductivity, watt/(cm)(°C)
30	2.155
400	0.666
748	0.384

The results are in fair agreement with other reported measurements on BeO of comparable density.

Oak Ridge⁵ obtained qualitative results on the resistance of hot-pressed BeO ceramics to corrosion by liquid sodium at 1210 to 1500°F. It was concluded that the purity and density of the BeO shapes and the flow velocity of the contacting sodium greatly influenced the measured loss of BeO by corrosion and erosion. Under exposure to slowly flowing sodium in the reported Aircraft Reactor Experiment (ARE), the corrosion resistance of commercial-BeO moderator blocks was deemed satisfactory.

(J. F. Quirk)

Solid Hydrides

Interest in the hydride-moderator-development program continues to be centered in zirconium and niobium hydride. The various experimental programs have as objectives the determination of physical, chemical, and mechanical properties of these materials.

Zirconium Hydride

Structural studies of ZrH_{1.92} conducted by Yakel,⁶ when both low- and high-temperature X-ray diffraction were used, have shown that the lattice symmetry is face-centered tetragonal at all temperatures. Indications are that the degree of axial splitting is temperature dependent, but this could not be determined without high-pressure equipment. There was no sig-

nificant change in composition of ZrH_{1.92} at the higher temperatures used. Figure 1 shows the variations of lattice parameters, axial ratio, and the cube root of the volume of the unit cell as functions of the absolute temperature.

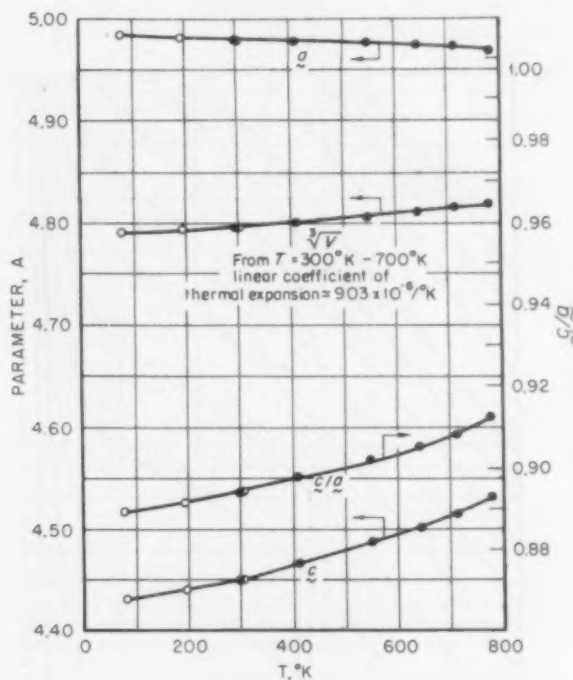


Figure 1—The variations of lattice parameters, axial ratio, and the cube root of the volume of the unit cell of ZrH_{1.92} as a function of the absolute temperature. O, low-temperature data. ●, high-temperature data. Data by Yakel.⁶

The hydriding of zirconium and Zircaloy-2 in monoisopropylbiphenyl has been investigated at Hanford. At temperatures between 250 and 400°C, concentrations of hydrogen in the metal ranging from the initial amount of 3 ppm up to 8700 ppm were obtained. The treatment lasted six days at a hydrogen partial pressure of 5 psi in a total pressure of 100 psi. Under these circumstances the hydriding process was shown to be dependent on both the hydrogen pressure and the temperature. No significant difference was observed in the performance of zirconium and Zircaloy-2.

Niobium Hydride

A study of low-pressure equilibria of the niobium-hydrogen system in the range 0.1 to 100 mm Hg pressure has been made at Battelle.⁷

These results have been combined with those previously reported for the niobium-hydrogen system to give a comprehensive coverage of the temperature range 100 to 900°C. Figure 2 is a logarithmic plot of the equilibrium pressure against composition for various temperatures.

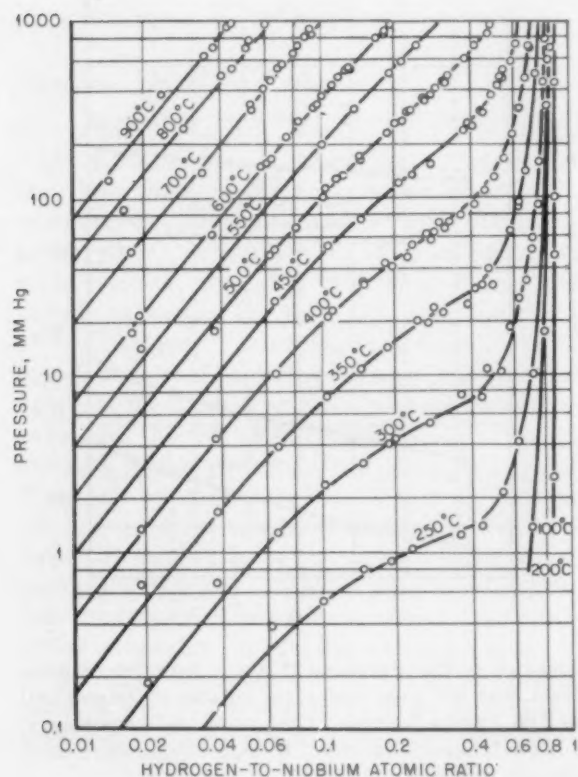


Figure 2—Logarithmic plot of isotherms in the niobium-hydrogen system. Data from Battelle Memorial Institute.⁷

An elevated-temperature X-ray study of the niobium-hydrogen system has also been made. Results obtained with specimens containing 0.097, 0.24, and 0.54 hydrogen-to-niobium ratios at room temperature to 400°C have outlined the boundary of a two-phase region. The data are shown in the phase diagram presented in Fig. 3. Several isobars for the system are also shown. From these data, the upper limit of the two-phase region was determined to be at about 150°C and at an equilibrium pressure of less than 0.1 mm Hg. Heats of solution for the niobium-hydrogen system, which were calculated from the data obtained in this study, are given in Table II-2. (H. H. Krause)

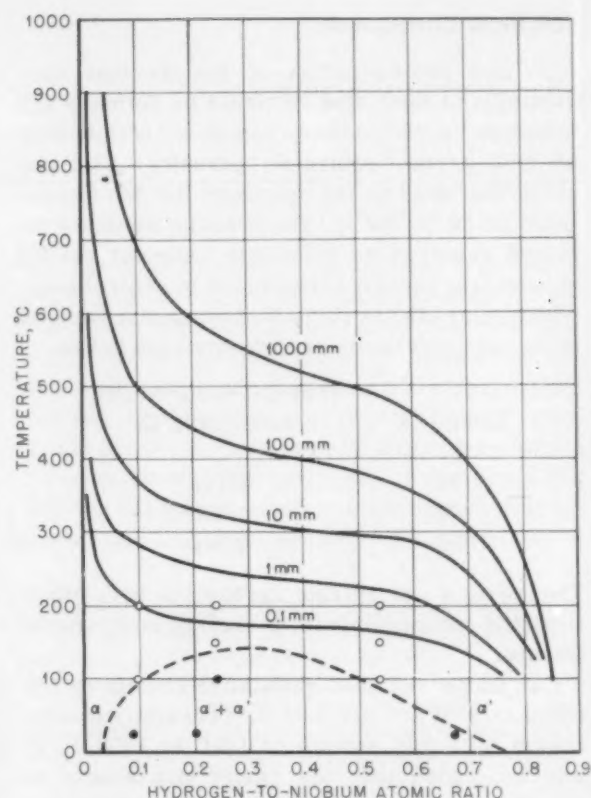


Figure 3—Phase diagram for the niobium-hydrogen system showing representative isobars. Determined by high-temperature X ray. O, single-phase region. ●, two-phase region. Data from Battelle Memorial Institute.⁷

Table II-2 HEATS OF SOLUTION FOR THE NIOBIUM-HYDROGEN SYSTEM*

Hydrogen-to-niobium ratio	Constants for log $P = A/T + B$		Heat of solution, cal/mole
	A	B	
0.01	-3480	4.83	15,900 ± 200
0.02	-3590	5.49	16,400 ± 130
0.05	-3570	6.17	16,400 ± 110
0.10	-3690	6.80	16,900 ± 120
0.20	-3950	7.55	18,090 ± 42
0.30	-4150	8.03	18,990 ± 47
0.40	-4300	8.40	19,700 ± 120
0.50	-4420	8.77	20,200 ± 140
0.50	-4320	8.90	19,800 ± 290
0.70	-4180	9.30	19,100 ± 1900

* Data from Battelle Memorial Institute.⁷

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Dispersion-control Materials

A special report (*Nucleonics*)¹ has been issued which includes some general information on design philosophies, control problems, and control rods. Corrosion resistance, mechanical properties, and costs of control-rod materials are given for some dispersion materials. Also, control-rod materials in use on many existing reactors are listed. Included are Boral (B_4C in aluminum), boron in titanium, and rare-earth oxides in stainless steel. (G. W. Cunningham)

Control-rod Alloys

Hafnium

From the results obtained by the Bureau of Mines,² it appears that Kroll-process sponge hafnium is suitable for reactor applications. Table III-1 compares typical mechanical properties obtained recently and at the beginning of the project with military specifications. All specimens were in either an annealed or hot-worked condition.

Table III-1 TENSILE PROPERTIES OF SPONGE HAFNIUM*

	Tensile strength (min), psi	Yield strength (min), psi	Elongation in 2 in. (min), %
Military Specification Mil-H-2103.	57,000	30,000	19
Typical at beginning of project	74,000	34,000	4-11
Obtained as of June 5, 1958	85,000	38,000	23

*Data from Bureau of Mines.²

Silver-Indium-Cadmium Alloys

Bettis³ is studying methods of improving the silver-15 wt.% indium-5 wt.% cadmium alloy in two respects: (1) the aqueous corrosion resistance of the alloy, which is affected adversely

when the water contains more than 0.3 ppm oxygen, and (2) the creep properties.

The corrosion resistance of the silver-indium-cadmium alloy is restored immediately upon replacement of high-oxygen water with normal water.

Creep resistance can be improved by coarsening the grain size, introducing a second phase by adding tin or increasing indium content to 18 wt.%, or by using powder alloys.

Knolls irradiated eight Instron tensile specimens of the silver-15 wt.% indium-5 wt.% cadmium alloy to a nominal exposure of 2×10^{21} n/cm² at 500°F. A summary of the available results is given in Table III-2. In addition, an

Table III-2 MECHANICAL PROPERTIES OF IRRADIATED SILVER-INDIUM-CADMIUM ALLOY*

Exposure, n/cm ²	2.11×10^{21}	2.11×10^{21}
Irradiation temperature, °F	500	500
Test temperature, °F	Room temp.	600
Yield strength (0.2% offset), psi	27,500	15,500
Ultimate tensile strength, psi	50,000	15,500
Elongation in 1.1 in., %	50.0	24.5
Reduction in area, %	53.5	32.5

*Data from Knolls Atomic Power Laboratory.

Instron sample which had been irradiated to 2.38×10^{21} n/cm² at 500°F was creep-rupture tested under a stress of 4000 psi at 600°F. The specimen failed in 31.3 hr with an elongation of 27.8 per cent.

Rare-earth Alloys

The Bureau of Mines⁴ has investigated gadolinium alloys of stainless steel and titanium which were prepared by arc-melting techniques. Some segregation was observed among alloys containing more than 20 wt.% gadolinium. Corrosion data are presented in Tables III-3 and III-4. Both stainless-steel- and titanium-base alloys containing less than 25 wt.% gadolinium exhibited excellent corrosion resistance in 680°F water and 750°F steam.

The hardness and brittleness of stainless-steel alloys increased with increasing gado-

Table III-3 CORROSION OF STAINLESS-STEEL-GADOLINIUM ALLOYS*

Gd, wt.%	Total test time in 680°F water, days	Weight gain, mg/cm ²	Total test time in 750°F steam, days	Weight gain, mg/cm ²
0.4	224	2	105	1
1.1	224	1	105	4
1.8	308	4	126	11
3.2	224	2		
11.0	196	30	112	12
22.0	28	22	21	10
29.5	14	-262†	7	82
32.5	14	-22†	7	-774†
34.0	28	17	21	6
35.0	14	-70†	7	-16†
43.0	14	226	14	102†

*Data from Bureau of Mines.⁴†Discontinued where gain in weight exceeded 100 mg/cm² or excessive weight loss occurred.

Table III-4 CORROSION OF TITANIUM-GADOLINIUM ALLOYS*

Gd, wt.%	Total test time in 680°F water, days	Weight gain, mg/cm ²	Total test time in 750°F steam, days	Weight gain, mg/cm ²
1.9	308	8	126	16
7.0	308	5		
9.3	252	22	126	33
20.0	196	82	112	84
24.0	28	6	21	48
29.0	28	87	21	87
35.0	28	52	14	117†
40.0	28	59	14	117†
46.0	28	43	14	112†
49.0	14	534†	7	182†
52.0	14	106†	7	143†
55.0	14	310†	7	275†
60.0	14	311†	7	326†

*Data from Bureau of Mines.⁴†Discontinued where gain in weight exceeded 100 mg/cm² or excessive weight loss occurred.

linium content. Stainless-steel-base alloys containing up to 10 wt.% gadolinium could be readily rolled at 850°C, whereas those containing up to 35 wt.% gadolinium showed evidence of workability when sheathed in stainless steel and rolled at 1000 to 1250°C. Most of the titanium-base alloys were hot-rolled at 850°C without difficulty. Fabrication data are shown in Tables III-5 and III-6.

Preparation of stainless-steel- and titanium-base europium alloys was complicated by the tendency of the europium to volatilize during

Table III-5 FABRICATION DATA FOR STAINLESS-STEEL-GADOLINIUM ALLOYS*

Gd, wt.%	Reduction in area, %	Rockwell hardness		Remarks
		As-cast	As-rolled	
1.8	85	B98		Button readily rolled to sheet
3.2	85	B87		Button readily rolled to sheet
10.0	85	C27		Button readily rolled to sheet
13.0		C36		Broke in fabrication
19.0		C38		Broke in fabrication
22.5	45	C37	B96	Rolled well; sheath leaked and surface oxidized
35.0	57.5	C48		Rolled, but very brittle
40.0		C56		Broke in fabrication
43.0				Welded to sheath

*Data from Bureau of Mines.⁴

Table III-6 FABRICATION DATA FOR TITANIUM-GADOLINIUM ALLOYS*

Gd, wt.%	Reduction in area, %	Rockwell hardness		Remarks
		As-cast	As-rolled	
2.7	59		C22	Fabricated to 0.049-in. sheet without difficulty
4.9		B89		Not fabricated
9.5				Fabricated to sheet
15.5		B87		Not fabricated
20.0				Rolled to sheet
23.0	70	B74	B79	Rolled readily
27.0	60	B69	B79	Rolled readily
35.0	69	B77	B84	Rolled readily
52.0	68	B82	B75	Rolled readily

*Data from Bureau of Mines.⁴

partial-vacuum arc melting. The maximum amount of europium retained in any of the alloys was 0.5 wt.%. Attempts to improve europium retention by melting under higher argon-helium pressures and by adding the europium in a master alloy with gadolinium were unsuccessful.

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Corrosion

Zirconium

Investigations concerned with the air oxidation of zirconium-base materials, crevice corrosion, effect of cold work, and quality-control corrosion tests are being conducted at several laboratories. However, two subjects are receiving major attention: (1) the stringer type corrosion phenomenon and (2) the hydrogen pickup by Zircaloy compositions during exposure to high-temperature water.

Studies at Bettis¹ have shown that, to maintain corrosion resistance of vacuum-melted Zircaloy-2, heat-treatments in the beta or alpha-plus-beta range must be followed by a rapid cooling rate (790°F/min) through the alpha-plus-beta phase. Slower cooling produces unacceptable corrosion rates, as measured by the standard 14-day test in 750°F, 1500 psi steam.

One of the controlling factors in the corrosion of Zircaloy compositions appears to be the distribution of alloying and impurity elements. During beta heat-treatments, diffusion rates are great enough to cause homogenization. However, at the start of the beta to alpha-plus-beta transformation, partitioning will be initiated by diffusion of elements to the phase having the greater solubility and will continue up to saturation. At the completion of transformation, diffusion rates are too slow for homogenization. As a result, there are local areas which are different in composition from the matrix. A rapid cooling rate suppresses diffusion and retains the homogeneous distribution of alloying elements that existed in the beta phase. A slow cooling rate, however, promotes the precipitation of intermetallic compounds along grain boundaries and in colonies, a condition which favors high corrosion rates.

The hydriding of zirconium and zirconium alloys in pressurized-water systems continues to be of primary concern. A critical review of the literature dealing with the conditions under which hydriding did and did not occur has

recently been completed by Hanford.² Other work at Bettis³ and Knolls⁴ has shown that Zircaloy-2—containing nickel additions hydrided in proportion to the amount of nickel added. Copper and iron additions had little or no effect on the hydrogen pickup; that is, the alloys exhibited no more than the usual amount of hydrogen pickup associated with corrosion in high-temperature water.

The role of hydrogen in the high-temperature corrosion of zirconium has recently been the subject of considerable investigation at Harwell.⁵ In these studies, zirconium-base materials were cathodically polarized during corrosion in water at 325°C. Current densities employed were 0.15 ma/cm², which corresponds in hydrogen discharge to a corrosion rate of 30 mg/(cm²)(month). It is suggested that hydrogen, on its way through the oxide film or on arrival at the metal-oxide interface, is able to hasten cracking of the protective film and thus increase the rate of corrosion. One possibility is that the presence of hydrogen may, in some way, increase the diffusion of undesirable ions which cause rapid film growth and subsequent cracking. Another suggestion is that the hydrogen may embrittle the oxide film, thus encouraging cracking. The most likely effect of the entrance of hydrogen into the metal itself seems to be the production of a separate hydride phase which causes volume changes and cracking of the protective films.

The scaling behavior of zirconium at high temperatures is the subject of investigations at the Case Institute of Tech.⁶ It is known that zirconium which has been oxidized in air at temperatures between 600 and 1300°C exhibits several interesting phenomena: (1) a change in color and porosity of the scale, (2) a distinct change in scaling rate (breakaway), and (3) lateral surface growth of the specimens. However, when iodide zirconium was exposed in pure oxygen, none of these effects was observed. This result led to the formulation of a proposed mechanism of scaling based on the presence of a ZrN layer between the metal and oxide and coherent with the metal lattice.

Growth is attributed to conversion of the ZrN layer to ZrO_2 , leading to a surface stress which, in conjunction with the stress arising from the formation of primary ZrO_2 , is sufficient to cause plastic deformation of the underlying metal. This growth is thought to rupture the protective scale which, in turn, leads to the observed change in scaling rate and color of the scale. However, in the light of recent work, the above mechanism appears to require some qualifications since growth has been noted for impure grades of zirconium exposed to pure oxygen. Although it has been shown that gaseous impurities in the metal, such as oxygen and nitrogen, do alter scaling rates in air, the data also indicate that observed growth of impure material in oxygen is not a function of these gaseous impurities. It now seems necessary to study the effects of other impurities in the metal.

Another problem which is receiving attention is that of quality-control corrosion tests. Bettis¹ found that a duplex steam-water test will reveal iron contamination of Zircaloy-2; an exposure of either one or two days in 680°F water, followed by two days in 750°F steam, was proposed. One drawback of this test is that it results in a higher rate of hydrogen pickup than is encountered in the standard steam test. (W. K. Boyd)

Stainless Steel

Considerable effort continues to be directed toward obtaining an understanding of the mechanism of the chloride stress-corrosion cracking of stainless steel. Oak Ridge⁷ reports that pre-filming of type 347 stainless steel in chloride-free uranyl sulfate-copper sulfate HRT core solution prevents cracking of materials subsequently exposed to the same solution containing 50 ppm chloride. Samples which were not prefilmed cracked within 400 hr. It is interesting to note, however, that this prefilming treatment was not effective in preventing stress-corrosion cracking of similar samples exposed at 260°C to water containing 10 ppm chloride. Sodium sulfite additions were found to prevent cracking in 50 ppm chloride water at 260°C. However, work at Bettis⁸ has indicated that, when the chloride level was increased to 500 ppm, cracking was observed even in the presence of residual sodium sulfite.

In other work, Staehle et al.,⁹ report that type 347 stainless steel is susceptible to cracking in 400°F chloride-containing water under

vapor condensate conditions. Threshold stress levels of less than 2,000 psi will cause cracking in water containing as low as 50 ppm chloride. Increasing the oxygen content of the water was found to increase the incidence of cracking, while an atmosphere of 100 per cent hydrogen or nitrogen eliminates cracking. (W. K. Boyd)

Niobium

Improvements in oxidation resistance were also effected by ternary additions of chromium-cobalt, chromium-nickel, iron-cobalt, and iron-nickel to niobium, according to recent Fansteel information by Michael.¹⁰ In air-oxidation tests at 1090°C, improvements up to twentyfold were obtained through the above additions. The best alloy from this work is niobium-13 wt.% chromium-9 wt.% cobalt which had a weight gain at 1090°C approximately ten times that of 80 wt.% nickel-20 wt.% chromium. The improvement is apparently due to formation of the protective spinel type scale, $CoCr_2O_4$.

Binary and ternary additions of titanium and zirconium to niobium were investigated at Ohio State University.¹¹ These additions improve the oxidation resistance by the formation of complex niobate scales, although the improvements are not as great as those effected by titanium-aluminum and chromium-cobalt additions. (W. D. Klopp)

Molybdenum

Protection of molybdenum from air oxidation by electroplating with chromium and nickel has been studied by Couch et al.¹⁴ Protection for periods up to 500 hr at 1100°C and 1200 hr at 980°C was possible when the applied coatings were 8 mils thick. The coating consisted of 1 mil of chromium overlaid with 7 mils of nickel. Interdiffusion of nickel and molybdenum and the formation of a brittle MoNi intermetallic layer were apparently retarded by the presence of chromium at the molybdenum surface. Pre-heating the plated molybdenum in an inert atmosphere to homogenize the coating or peening to close voids in the coating provided no further improvement in the subsequent resistance to air oxidation. The oxidation resistance of these electroplated samples was at least as good as that of samples protected by sprayed aluminum-chromium-silicon coatings for which lives of up to 500 hr at 1000°C were obtained. (W. D. Klopp)

Tantalum

The reaction of tantalum with air and oxygen has been studied in recent years over the temperature^{10,15-20} range 50 to 1090°C. The reactions are very similar to those of niobium, its lower-melting sister element. At low temperatures, the reactions of tantalum with air and oxygen are similar. No color change is noted after short exposures up to 200°C, but above this temperature a blue tarnish film forms, changing to black at higher temperatures. Up to 320°C, the reactions follow the logarithmic rate law [$W = K \ln(t + 1)$]. From 320 to 350°C, the reaction follows roughly the cubic rate law ($W^3 = Kt$), and from 350 to about 500°C, the parabolic rate law ($W^2 = Kt$) is followed. Above 500°C, the black adherent oxide film breaks down, forming a white porous scale which is no longer protective. The oxidation reaction in this temperature range follows the linear rate law ($W = Kt$) and is slightly faster in pure oxygen than in air. The rates are close to those of pure niobium.

The effects of alloying on the oxidation resistance of tantalum have recently been studied by Michael.¹⁰ As with niobium, zirconium and titanium additions improved the oxidation resistance of tantalum. Binary additions of iron, cobalt, and nickel also effected improvements, whereas additions of molybdenum and tungsten did not. Combinations of elements which oxidize preferentially or which form spinel type scales were found to be most effective. The best compositions, as determined from 16-hr exposures in air at 1090°C, were tantalum-14 wt.% titanium-9 wt.% cobalt or nickel, and tantalum-7 wt.% chromium-5 wt.% cobalt or nickel. (W. D. Klopp)

Nickel-base Alloys

1. *Aqueous Corrosion.* In recent work at Oak Ridge,²¹ the corrosion resistance of nickel-base alloys in corrosive media such as aqueous uranyl sulfate and water containing chloride ions has been evaluated. It was found that the resistance to stress-corrosion cracking in water containing chlorides is generally better in alloys with higher nickel content. For example, after 500 hr in 300°C water containing 100 ppm chloride ion, Incoloy (32.5 per cent nickel) exhibited some stress-corrosion cracking, whereas Nionel (40 per cent nickel) did not. An electroless nickel plate on carbon steel was found to afford pro-

tection against chloride stress-corrosion cracking. In an 0.17M uranyl sulfate solution, Nionel and Incoloy were inferior in general corrosion resistance to several stainless-steel grades tested. All stainless as well as higher alloy steels tested exhibited very good resistance to corrosion by 300°C water.

2. *Oxidation.* Work is continuing at the National Bureau of Standards on the evaluation of the oxidation resistance of two additional nickel-base alloys, INOR-8 (70 wt.% nickel-16 wt.% molybdenum-7 wt.% chromium-5 wt.% iron + tungsten, silicon, cobalt, manganese)²² and a 77 wt.% nickel-13.5 wt.% molybdenum-6 wt.% chromium-1.75 wt.% titanium-1 wt.% aluminum-1 wt.% columbium alloy²³ which is referred to as a "nickel-molybdenum alloy." Oxidation resistance of the alloys was evaluated at temperatures from 1500 to 2000°F under stress. While elevated-temperature strength of the nickel-molybdenum alloy was markedly superior to that of INOR-8, the rate of oxidation of INOR-8 was considerably less. Both alloys were inferior in oxidation resistance to Hastelloy R235 and Hastelloy W, evaluated previously. The activation energies of the oxidation reaction were calculated to be 51,400 cal/mole for INOR-8 and 43,280 cal/mole for the nickel-molybdenum alloy. (E. S. Bartlett)

Iron-Chromium-Aluminum Alloys

The Naval Research Laboratory²⁴ studied the resistance of iron-chromium-aluminum alloys to oxidizing atmospheres containing V_2O_5 . Although these alloys are characteristically weak at elevated temperatures and present difficulties in fabrication, their resistance to catastrophic V_2O_5 attack in the temperature range from 1600 to 1800°F renders them worthy of consideration for applications where this atmosphere is expected. Chromium content was varied from 0 to 26 wt.% and aluminum content from 0 to 14 wt.% in the alloys studied. It was found that whereas higher aluminum contents improved the resistance to V_2O_5 attack, the higher aluminum-to-chromium ratio rendered the alloys more susceptible to fissuring in straight oxidizing atmospheres. (E. S. Bartlett)

Corrosion by Liquid Metals and Fused Salts

Fused-salt and liquid-metal corrosion studies in connection with the Molten Salt Reactor Program at Oak Ridge are currently quite ex-

tensive.²⁵ Inconel has been tested in 1000-hr thermal-convection loop tests with eight different fluoride salts (based on constituents such as NaF, BeF, and LiF, and containing ThF₄ and UF₄); peak temperatures were in the 1200°F range, with temperature cycles of 125 and 170°F. None of the salts attacked the Inconel severely; hot-zone attack to a depth of 2 mils was about the maximum found. Experiments were also run to evaluate the susceptibility of Inconel and INOR-8 (a nickel-molybdenum alloy) to carburization in a high-temperature liquid environment. The data listed in Table IV-1 (for static capsule tests with graphite inserts) indicate that INOR-8

During the past year, Atomics International carried out a corrosion program²⁶ to find ferritic steels to replace austenitic steels for sodium containment at the 1000°F level. Test steels such as 2½ wt.% chromium-1 wt.% molybdenum; 5 wt.% chromium-½ wt.% molybdenum-titanium, 5 wt.% chromium-½ wt.% molybdenum-silicon, and 9 wt.% chromium-1 wt.% molybdenum were exposed to 1000°F sodium in loop apparatus (fabricated of type 304 stainless steel) for 1000 and 2000 hr; temperature cycles were 150°F and sodium flow rates were 2 and 5 ft/sec. It was found that the 2½ wt.% chromium steels decarburized significantly in the presence

Table IV-1 RESULTS OF CARBURIZATION TESTS OF INCONEL AND INOR-8 SPECIMENS EXPOSED TO SODIUM AND TO FLUORIDE SALTS AT 1500°F FOR 100 HR*

Total carbon in as-received Inconel: 0.038 per cent

Total carbon in as-received INOR-8: 0.020 per cent

Test system	Alloy tested	Weight change, %	Total carbon content, %	Metallographic results
Graphite; sodium	Inconel	+0.50	0.60	Carburized to 10 to 12 mils, the first 1½ mils being a light band in which carbide particles were agglomerated
	INOR-8	+0.36	0.36	Carburized throughout; first 9 mils showed a heavy precipitate; carbide particles less dense in interior of specimen
Graphite; NaF-BeF ₂ -UF ₄ (53-46-1 mole %)	Inconel	+0.20	0.04	Attack to 2 mils in form of small subsurface voids; no carburization observed
	INOR-8	+0.30	0.01	Small stringers 0.5 mil deep along edge; fine scattered particle formation to a depth of 1 mil
Graphite; LiF-BeF ₂ -UF ₄ (62-37-1 mole %)	Inconel	-0.37	0.03	Attacked to a maximum depth of 2 mils in the form of small subsurface voids; no carburization observed
	INOR-8	+0.03	0.01	Particles found in the grain boundaries to a depth of 1.5 mils

*Oak Ridge National Laboratory data.²⁵

is more sensitive to carburization than Inconel, presumably because the molybdenum in the INOR-8 forms carbides more readily than does the chromium in Inconel. In addition to loop type corrosion studies, basic work in the chemistry of the fluoride-salt corrosion processes is continuing. For example, the preliminary data from an investigation of the activity coefficients of CrF₂ in NaF-ZrF₄ have been obtained.²⁵ Experimental equilibrium-ratio data yielded activity coefficients for CrF₄ of 0.284 and 0.181 at 850°C with respect to the solid and the supercooled-liquid standard states, respectively.

of the austenitic steel, regardless of the carbon content of the latter. Much better performers from the decarburization viewpoint were the 5 wt.% chromium-titanium steels; no advantage was gained by increasing the chromium above 5 wt.%. Although increasing the chromium from 2½ to 5 wt.% inhibited decarburization, it was mentioned that higher fluid thermal cycles (above 150°F) may lead to decarburization of even the higher chromium steel.

The effects of high-temperature sodium on zirconium in the 1000°F range have also been investigated²⁷ extensively; the most deleterious effect is the lowering of fatigue life owing to

factors such as surface-oxide formation, absorption of hydrogen, and exaggerated grain growth. Zirconium is oxidized by exposure to oxygen-containing sodium, but Zircaloy-2 is sensitive to a lesser degree. When the sodium is appropriately gettered, Zircaloy-2 (and niobium, which is also subject to oxidation in sodium) is not affected by prolonged exposure. Experiments in which zirconium was immersed in sodium in contact with carbon or nitrogen at 1000°F displayed no evidence of carbide or nitride formations. Type 304 stainless steel² exposed to nitrogen-covered sodium displayed a 1-mil nitride case at the sodium-nitrogen interface after 1600 hr.

The containment of the following uranium-bearing molten metals has recently been studied:²⁸ (1) the uranium-chromium eutectic (by yttrium at 900 and 925°C), 5 wt.% uranium-95 wt.% bismuth (by niobium at 800°C and by yttrium at 900°C), and the aluminum-thorium-uranium eutectic (by tantalum at 1000°C and by yttrium at 700°C). The most compatible combination was the uranium-chromium eutectic in yttrium. After exposures for as long as 1000 hr, attack could not be observed. Yttrium also resisted attack fairly well by the uranium-bismuth alloy and the aluminum-thorium-uranium eutectic; for example, in the former case, 1000 hr of exposure produced only 35 mils of attack. The niobium and the tantalum in the cases mentioned above failed after 100 hr.

A recently published paper²⁹ presents a summary of findings from extensive research carried out by Kammerer et al., on the inhibition of solution mass transfer of pure iron and steels by heavy-liquid metals (bismuth and lead-bismuth alloys) through small additions of zirconium and titanium. The temperature range of interest was 500 to 800°C. These additives react with the liquid-contacting surface to form adherent deposits of ZrN, TiN, or TiN + TiC. These deposits are inert to liquid bismuth and lead-bismuth (and, on the basis of results from other investigations, probably to mercury and lead). The rate of deposit formation is probably controlled by the activity of the nitrogen in the container material. In a polythermal flow system, the additives appear to have a suppressive effect on cold-zone mass transfer. Apparently, a stabilization of a supersaturated solution of iron in the cold zone is involved. In the mechanism postulated, a monomolecular layer of

zirconium, for example, would coat the surfaces of nucleated-iron particles in the cold zone and, as a result, the coated particles would be prevented from forming agglomerates massive enough to fall out of the flow stream.

(J. H. Stang)

Zirconium-Water Reactions

The zirconium-water reaction which might occur in a reactor loss-of-coolant accident or nuclear runaway has been qualitatively analyzed recently by Knolls.³⁰ No new experimental data are presented, but the concepts evolved are of interest. The basic concept is that, at some point in the course of the reaction, the fundamental nature of the reaction becomes governed by external hydrodynamic (and heat transfer) considerations, rather than by chemical considerations; i.e., the reaction rate becomes limited by mass and energy transfer. It is postulated that this occurs where the temperature is above the melting point but far below the vaporization point of the metal. On the basis of this concept, a previous analysis of the loss-of-coolant accident is reviewed and the analysis extended to the case of the nuclear runaway. The assumed temperature dependence of the reaction rate implies a less severe reaction prior to vaporization of the metal than had been assumed heretofore. Additional emphasis is placed, in this qualitative analysis, on the importance of the physical configuration and water availability in any evaluation of the possible extent of metal-water reaction in a postulated nuclear-reactor failure.

Bettis³¹ recently reported a calculational method for determining the effect of external quenching of fuel-element assemblies on the extent of metal-water reaction and the maximum temperature during a temperature excursion subsequent to a postulated loss-of-coolant accident. However, no new zirconium-water reaction-rate data are reported. The method depends upon having experimental quenching data available or being able to estimate it. The calculational method may be used whenever simultaneous core cooling, reactor heat generation, and the possibility of a zirconium-water reaction exist.

(A. W. Lemmon, Jr.)

Selected Mechanical Properties of Cladding and Structural Materials

Zirconium and Zirconium Alloys

The effect of heat-treatment on the tensile properties of a high-strength zirconium-5 wt.% molybdenum-2 wt.% aluminum alloy has been investigated at Nuclear Metals.^{32,33} The effects of aging time and temperatures on the room-

Alloys containing from 2.1 wt.% through 19.0 wt.% niobium exhibited increased hardness after aging for 8 hr at 300°C. The decrease in hardness observed for the zirconium-6.0 and 9.4 wt.% niobium after aging at 500°C for 44 hr is reported to have occurred as the result of the diffusion process in the lower-niobium alloy which was sufficiently rapid to cause softening.

Westinghouse reports that zirconium-niobium binary or ternary zirconium + niobium + X com-

Table IV-2 ROOM-TEMPERATURE TENSILE PROPERTIES OF AGED ZIRCONIUM-BASE ALLOYS*

Composition, wt. %	Aging time and temperature	Tensile strength, 1000 psi	Yield strength (0.2% offset), 1000 psi	Elongation in 1 in., %	Elastic modulus, 10 ⁶ psi
Zr-5 Mo-2 Al	1/2 hr at 500°C	200	184	6.88	16
Zr-5 Mo-2 Al	96 hr at 600°C	127	110	8.76	15

* Data from Nuclear Metals.³²

temperature tensile properties are shown in Table IV-2. The alloys were solution treated 1 hr at 760°C, water quenched, and aged as shown in the table.

This alloy was coextruded with uranium to produce a clad thickness of 0.040 in. The clad specimen showed only slight to moderate distortion and only a small increase in length after 1100 thermal cycles between room temperature

positions (where X was palladium, platinum, or iron) exhibited a marked response to heat-treatment.³⁵

Studies of the deformation process at room temperature in zirconium have been completed at Nuclear Metals.³⁶ The observed slip system was the (1010) plane and (1210) direction and had a critical resolved shear stress for slip in compression of about 0.65 kg/mm². Slip on the basal slip plane was not observed, although some crystals were favorably oriented for basal slip. Active twin planes identified as the (1012), (1121), and (1122) were observed. All twins observed were mechanical twins produced under stress and had a characteristic "cry" upon formation. (F. R. Shober)

Table IV-3 HARDNESS OF ZIRCONIUM-NIOBIUM ALLOYS AFTER HEAT-TREATMENT*

Composition (bal. zirconium), wt. %	Brinell hardness after indicated heat-treatment		
	Quench from 650°C	8 hr at 300°C	44 hr at 500°C
2.1 Nb	185	205	
6.0 Nb	320	375	290
9.4 Nb	290	340	270
11.3 Nb	295	350	330
19.0 Nb	174	223	202
40.0 Nb	190		
60.0 Nb	212		

* Data by Bichkov et al.³⁴

and 500°C. The effect of aging time and temperature on the hardness of a series of zirconium-niobium alloys is reported by Bichkov et al.³⁴ As shown in Table IV-3, as-quenched hardness was increased by low-temperature heat-treatment, presumably by aging.

Zircaloy-2 and -3

The mechanical properties, particularly the creep properties, of Zircaloy-2 and -3 have been investigated at Bettis.³⁷⁻³⁹ These materials were tested in the following conditions:

1. As received; approximately 15 per cent cold worked by swaging.
2. Base anneal; annealed in vacuum 20 hr at 750°C.
3. Base anneal plus 10 per cent cold work; annealed in vacuum 20 hr at 750°C, machined, cold swaged 10 per cent.
4. Beta quenched; annealed at 1000°C for 4 hr in Vycor tube (argon atmosphere), water

Table IV-4 TENSILE PROPERTIES OF ZIRCALOY-2 AT ROOM TEMPERATURE, 250°C AND 350°C*

Zircaloy-2	Test temperature, °C	Yield strength (0.2% offset), 1000 psi	Tensile strength, 10 ³ psi	Elongation, %	Reduction in area, %
As received	Room temp.	83.0	95.0	8.0	30
As received	250	55.2	60.8	9.9	46
As received	350	51.0	53.6	8.0	51
Base anneal	Room temp.	44.0	68.8	30.0	48
Base anneal	250	19.4	36.0	25.0	54
Base anneal	350	16.3	30.9	29.6	75.5
Base anneal	Room temp.	83.0	95.0	9.0	44
+ 10% cold work	250	46.6	51.0	8.95	55
+ 10% cold work	350	41.0	42.6	7.9	68
Beta quenched	Room temp.	77.8	87.5	9.0	42
+ 10% cold work	250	57.8	61.8	8.4	64
+ 10% cold work	350	52.8	57.2	7.6	66

* Data from Bettis.³⁷

Table IV-5 TENSILE PROPERTIES OF ZIRCALOY-3 AT ROOM TEMPERATURE, 250°C AND 350°C*

Zircaloy-3	Test temperature, °C	Yield strength (0.2% offset), 1000 psi	Tensile strength, 10 ³ psi	Elongation, %	Reduction in area, %
As received	Room temp.	88.2	98.4	8.0	34
As received	250	55.8	59.6	9.4	50
As received	350	43.6	47.6	13.9	62
Base anneal	Room temp.	44.8	71.8	29.0	42
Base anneal	250	16.4	33.8	30.6	64
Base anneal	350	14.1	25.6	35.4	75.5
Base anneal	Room temp.	90.2	102.0	8.3	35
+ 10% cold work	250	45.0	47.6	11.9	48
+ 10% cold work	350	35.0	36.1	8.67	64
Beta quenched	Room temp.	103.0†	110.0†	10.5†	46†
+ 10% cold work	250	58.2	60.1	8.3	63.5
+ 10% cold work	350	50.8	52.8	6.9	66

* Data from Bettis.³⁷

† Average of two tests.

quenched, cold swaged to 10 per cent reduction prior to machining.

Zircaloy-2 was found to have much better creep properties over the 200 to 400°C range than Zircaloy-3, but the tensile properties were quite similar. The differences in creep behavior were magnified at the lower secondary-creep rate and higher temperatures. Test results for the tensile and creep properties are shown in Tables IV-4 through IV-7. The tensile properties of vacuum-melted³ Zircaloy-2 tested in the longitudinal and transverse directions to rolling are given in Table IV-8.

The creep properties of atmosphere melted, extruded, and annealed Zircaloy-2 were determined in the range 600 to 800°F. The increased rupture strength of the atmosphere melted Zircaloy-2, as compared with vacuum-melted material, is more evident at low temperature than at elevated temperatures. The results are given in Table IV-9. The tensile and yield strengths of fusion-welded Zircaloy-2 after being annealed for 1 hr at 800°C are slightly higher than those of wrought Zircaloy-2. A reduction in ductility of the fusion-welded Zircaloy-2 was evident, the elongation being

Table IV-6 CREEP PROPERTIES OF ZIRCALOY-2 AT 250 AND 350°C*

Zircaloy-2	Test temp., °C	Stress, 1000 psi	Plastic strain on loading, %	$\dot{\epsilon}$		Transition point		Rupture		Reduction in area, %
				ϵ_0 , %	in./ (in.) (hr)	ϵ , %	t, hr	ϵ , %	t, hr	
As received	250	59.0	0.27	1.02	8.3×10^{-4}	3.3	29.5	7.64	38.2	45.7
As received	250	56.0	0.23	1.23	7.1×10^{-5}	3.49	332	7.56	468	43.9
As received	250	53.0	0.25	0.80	1.2×10^{-6}	1.31+	3661+		†	
As received	250	50.0	0.08	0.375	5×10^{-7}	0.545+	3717+		†	
As received	350	51.3	0.50	0.95	2.3×10^{-2}	3.5	1.0	14.1	2	53.6
As received	350	45.0	0.26	0.76	3.5×10^{-3}	3.71	8.0	14.0	14.5	58.9
As received	350	41.0	0.05	0.52	5.0×10^{-5}	3.38	530	19.2	1087	57.0
As received	350	37.5	0.02	0.40	1.6×10^{-5}	4.0	2600	15.4+	3525+†	
As received	350	35.0	0.02	0.79	6.2×10^{-6}	2.13+	2135+		†	
As received	350	32.0	0.01	0.268	2.2×10^{-6}	0.96+	3160+		†	
Base anneal	250	32.0	4.67		5×10^{-2}			26.6	2.5	71.6
Base anneal	250	30.0	2.35	≈11	$\approx 2 \times 10^{-6} \ddagger$	11.2+	990+		†	
Base anneal	250	28.0	1.53	4.0	$\approx 2 \times 10^{-6} \ddagger$	4.6+	1740+		†	
Base anneal	250	26.0	1.859	2.55	10^{-7}	2.5+	800+		§	
Base anneal	350	27.0	5.0	{ 13.0	4.9×10^{-4}					
				{ 12.4	6.2×10^{-4}	18.4	87	21.55	88	66
Base anneal	350	25.0	3.39	6.7	2.3×10^{-5}	15.47+	3794+		†	
Base anneal	350	23.0	2.09		†	2.62+	162+		§	
Beta quenched	250	57.5	0.25	0.65	1.6×10^{-4}	2.8	68	4.9	69	54
+ 10% cold work	250	56.0	0.19	0.25	3.5×10^{-6}	0.76+	475+		†	
+ 10% cold work	350	51	0.06	0.71	1.9×10^{-4}	3.0	173	7.86	287	52
Base anneal + 10% cold work	250	46.0	0.08	0.48	1.15×10^{-3}	1.9	12	6.18	14	60

* Data from Bettis.³⁷

§ In test.

† Test stopped.

‡ Still in first-stage creep.

‡ Test ran in spring machines (estimate only).

Table IV-7 CREEP PROPERTIES OF ZIRCALOY-3 AT 250 AND 350°C*

Zircaloy-3	Test temp., °C	Stress, 1000 psi	Strain on loading, %	e, %		Transition point		Rupture		Reduction in area, %
				e ₀ , %	$\dot{\epsilon}$, in./in.(hr)	e, %	t, hr	e, %	t, hr	
As received	250	53.0	0.20	0.66	4.8×10^{-3}	3.25	5.1	10.47	8.4	50
As received	250	50.0	0.08	0.49	5.2×10^{-4}	3.55	55.0	13.7	105.5	52
As received	250	47.0	0.06		$\approx 4 \times 10^{-5}$			18.3	2680	44
As received	250	44.0	0.09	1.35	1.9×10^{-5}	4.085+	1500+		†	
As received	250	44.0	0.00	0.68	1.45×10^{-5}	5.5+	3330+		†	
As received	250	42.0	0.00	≈ 0.32	2.34×10^{-6}	1.07+	3100+		†	
As received	350	40.0	0.3	0.63	2.3×10^{-2}	3.68	1.3	13.74	2.8	58
As received	350	36.0	0.025	0.325	3.2×10^{-3}	3.65	20	21.3	31	60
As received	350	32.0	0.14	0.45	2.0×10^{-4}	2.84	120	8.4+	255+†	
As received	350	30.0	0.00	0.50	1.0×10^{-4}	2.7	218	35.5	707	66
As received	350	25.0	0.006	0.29	1.7×10^{-5}	3.04	1580	17.5+	4026+	
Base anneal	250	29.0	3.58	7.2	4.9×10^{-2}			30.6	3.5	62
Base anneal	250	25.0	2.08	7.68	3.4×10^{-3}	24.7	49	33.6	57	63
Base anneal	250	21.0	1.66	3.978	9.0×10^{-7}	4.16+	2100+		†	
Base anneal	250	22.0	1.76	7.04	9×10^{-7}	7.17+	1751+		†	
Base anneal	350	22.0	3.53	{ 9.63 6.53	{ 4.5×10^{-3} 7.8×10^{-3}	≈ 23.3	≈ 24	39.06	24.5	
Base anneal	350	22.0	9.5	10.3	1.8×10^{-2}	16.2+	3.3+		†	
Base anneal	350	18.0	1.48	{ 4.28 3.48	{ 5.2×10^{-4} 5.9×10^{-4}	≈ 25	≈ 310	50.2	370	73
					9.7×10^{-4}					
Base anneal	350	16.0	1.155	≈ 7.1	$\approx 1.6 \times 10^{-6}$		953+		†	
Beta quench	250	57.5	0.14	1.70	1.0×10^{-4}	3.8	200	8.37	253	
+ 10% cold work	250	51.5			$\approx 7 \times 10^{-6}$		120+		§	
+ 10% cold work	250	50.0	0.0		3×10^{-6}		389+		§	
+ 10% cold work	250	58.0	0.067	0.76	2.7×10^{-4}	2.2	52		58	
+ 10% cold work	350	45.0	0.0	0.6	4.6×10^{-4}	2.2	37	18.38	69	66
Base anneal	250	41.0	0.06	0.16	2.3×10^{-3}	1.0	3.5	7.3	6.0	
+ 10% cold work	250	36.0	0.00	0.25	3.5×10^{-5}	1.4	262	7.6	506	

* Data from Bettis.³⁷

† Test stopped.

‡ In test.

§ Test ran in spring machines (estimate only).

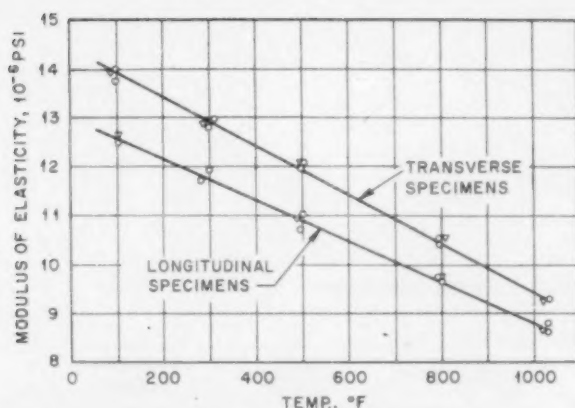


Figure 4—Dynamic modulus of elasticity of Zircaloy-2 between room temperature and 1000°F. Data from Knolls Atomic Power Laboratory.

Table IV-8 ROOM-TEMPERATURE TENSILE PROPERTIES OF VACUUM-MELTED ZIRCALOY-2*

	Longitudinal	Transverse
Tensile strength, 1000 psi		
Average	61	66
Maximum	69.3	80
Minimum	55.6	55.6
Yield strength (0.2% offset), 1000 psi		
Average	40.2	55.4
Maximum	44.5	69
Minimum	37.5	37.9

* Data from Bettis.³

approximately 50 to 60 per cent lower than that observed for wrought Zircaloy-2.

Data from 15 per cent cold-worked Zircaloy-2, in test at Battelle,^{12,13,40} indicate that the 10,000-hr creep strengths at 290, 345, and 400°C are 35,000, 25,000, and 15,000 psi, respectively. The dynamic modulus as determined at Knolls is shown in Fig. 4. The specimens were tested in the fully annealed condition and were taken from rolled plate both parallel and transverse to the rolling direction. There is a definite directionality effect on the modulus which becomes somewhat more pronounced with increasing temperature.

The effect of beryllium, as an alloying element, on the tensile properties of Zircaloy-3 has been investigated by Bettis.⁴¹ Alloys containing 0.05, 0.15, 0.35, and 0.70 wt.% beryllium were cast and fabricated by extruding. As-extruded bars had tensile and yield strengths as high as 91,000 and 54,000 psi, respectively, without serious loss of ductility. Further hot working apparently increases the ductility without an increase in strength. Tensile data obtained at 250°C indicate an increase in strength over both Zircaloy-2 and -3 when 0.35 wt.% beryllium is added to each. Table IV-10 shows the results.

The effect of hydrogen on Zircaloy-2 mechanical properties from room temperature to 1100°F has been investigated at Knolls.⁴² The results are given in Figs. 5 through 7. (F. R. Shober)

Table IV-9 STRESS-RUPTURE DATA FOR EXTRUDED AND ANNEALED ZIRCALOY-2 TESTED AT 600, 700, AND 800°F*

Temp., °F	Yield strength (0.2% offset), 1000 psi	Creep stress, 1000 psi	Rupture life, hr	Reduction in area, %	Minimum creep rate, in./in./hr	Time to reach indicated deformation, hr			
						10	5	2	1
600	18.9	32	0.716	67.5	19.2	0.13			
600	23.7	31	1.83	73.0	4.2	0.74	0.063		
600	21.5	30	15	70.3	0.165	3.05	0.083		
600	22.3	29.5	256	68.9	0.0105	10	0.108		
600	19.0	28.5	895	74.0	0.0013	886	0.225		
700	17.5	28	8.5	75.5	0.86	1.83			
700	16.6	27	57	67.5	0.25	16.6	1.5		
700	16.8	26.5	87	71.7	0.23	19.5	1.2		
700	18.6	25.5	389	67.5	0.045	119	17		
700	19.0	24.9	369	75.0	0.028	235	60	0.37	
700	18.0	24.8	719	70.7	0.030	238	62	0.16	
800	15.6	24	9	73.7	2.33	2.4	0.42		
800	17.0	21	38	73.7	0.64	11.5	3.9	0.3	
800	16.2	16	753	74.2	0.034	180	54	9.65	1.4
800		14.5			0.009	472	142	23	3.85

* Data from Bettis.³⁸

Table IV-10 TENSILE PROPERTIES OF ZIRCALOY-3 WITH BERYLLIUM ADDITIONS*

Zircaloy-3 plus wt. % beryllium	Yield strength (0.2% offset), 1000 psi		Tensile strength, 1000 psi		Elongation, % Uniform Total		Reduction in area, %
Room-temperature Zircaloy-3, as-extruded specimen, threaded rounds, 0.252 in. in diameter							
0.05	{ Top	40.1	70.4	14	18	34	
	{ Middle	38.7	73.4	16	19	36	
0.15	{ Top	40.4	74.4	15	19	34	
	{ Middle	46.8	76.8	11	13	30	
0.35	{ Top	42.0	77.0	12	17	34	
	{ Middle	43.2	81.0	14	18	28	
0.70	{ Top	54.8	90.8	12	15	30	
	{ Middle	68.0	92.3	3	3	6	
Extruded and hot-forged flats, $\frac{3}{8}$ - \times $\frac{1}{2}$ - \times 1-in. reduced section							
0.05	{ Top	49.0	75.3	18	30	41	
	{ Middle	39.7	75.3	20	28	43	
0.15	{ Top	58.7	75.5	18	27	32	
	{ Middle	56.5	75.5	19	26	71	
0.35	{ Top	40.2	77.7	21	28	44	
	{ Middle	42.8	72.6	22	30	41	
0.70	Top	53.3	89.0	16	19	27	
250°C tensile tests, Zircaloy-3, threaded round, 0.252 in. in diameter							
0.05	{ Top	23.3	44.0	31	61	31	
	{ Middle	25.0	42.5	30	52	28	
0.15	{ Top	23.6	40.5	34	45	27	
	{ Middle	24.1	43.7	28	49	28	
0.35	{ Top	28.7	45.0	15	18	7	
	{ Middle	27.2	49.5	26	49	23	

* Data from Bettis.⁴¹

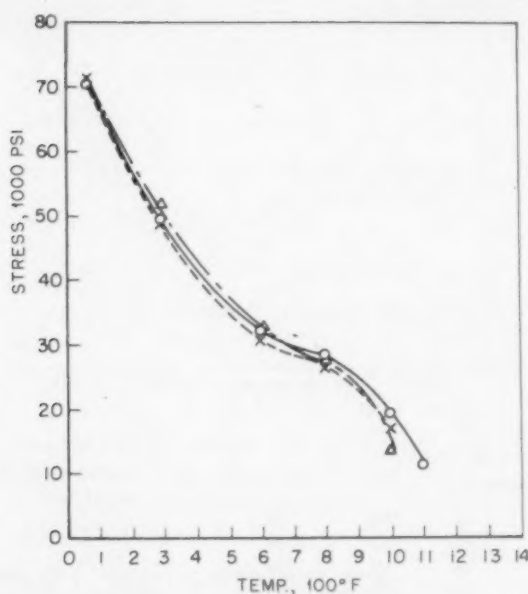


Figure 5—Ultimate tensile strength as a function of temperature and hydrogen content. For annealed Zircaloy-2 specimens removed longitudinally to the rolling direction. O, 12 ppm hydrogen. x, 150 ppm hydrogen. Δ, 500 ppm hydrogen. Data from Knolls Atomic Power Laboratory.

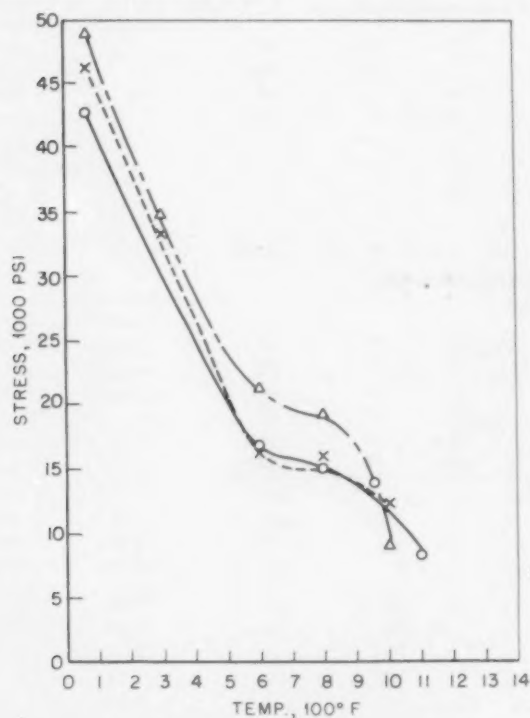


Figure 6—Yield strength (0.2% offset) as a function of temperature and hydrogen content. For annealed Zircaloy-2 specimens removed longitudinally to the rolling direction. O, 12 ppm hydrogen. x, 150 ppm hydrogen. Δ, 500 ppm hydrogen. Data from Knolls Atomic Power Laboratory.

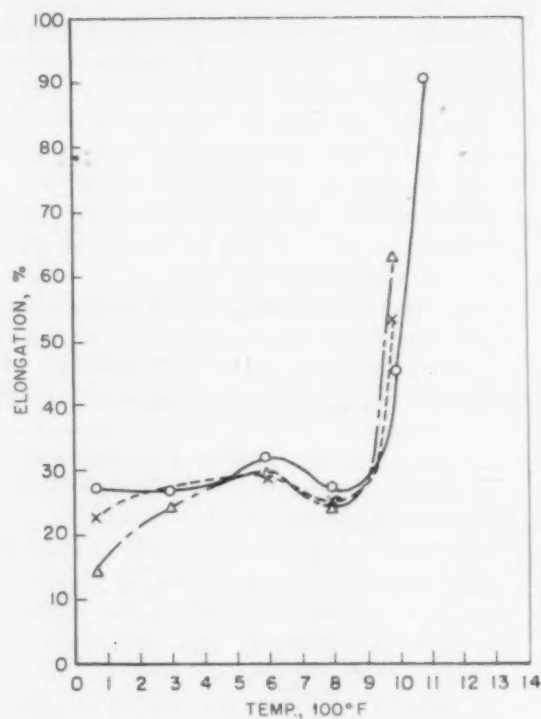


Figure 7—Per cent elongation as a function of temperature and hydrogen content. For annealed Zircaloy-2 specimens removed longitudinally to the rolling direction. O, 12 ppm hydrogen. x, 150 ppm hydrogen. Δ, 500 ppm hydrogen. Data from Knolls Atomic Power Laboratory.

Nickel-base Alloys

The mechanical properties of a nickel-base molybdenum alloy (nominal composition 17 wt.% molybdenum-7 wt.% chromium-5 wt.% iron-balance nickel, designated as INOR-8) are being investigated at Oak Ridge²⁵ to obtain basic data on the strength of INOR-8 required for design calculations. Aging INOR-8 for 500 hr at 1000, 1100, 1200, 1300, and 1400°F, respectively, had very little effect on its room-temperature tensile properties, as is shown in Table IV-11. The tensile and yield strengths of INOR-8, measured at room temperature, 1000, 1100, 1200, and 1300°F, are presented in Table IV-12. Also, relaxation tests (tests to study the plastic properties of a material) were made on INOR-8. In relaxation tests, a specimen is loaded to a fixed amount of strain, and that strain is maintained either by adding to the load or subtracting from it. The need to remove the load to maintain a fixed strain indicates that the material deformed plastically. The results

Table IV-11 ROOM-TEMPERATURE TENSILE PROPERTIES* OF INOR-8

Heat-treatment	Tensile strength, 1000 psi		(0.2% offset), psi		Elongation, %	
	Heat SP-19	Heat 8M-1	Heat SP-19	Heat 8M-1	Heat SP-19	Heat 8M-1
Annealed	114.4	117.1	44.7	51.9	50	39
Annealed and aged 500 hr at 1000°F	112	115.7	42.5	47.2	53	43
Annealed and aged 500 hr at 1100°F	112.6	114.5	44	48	51	43
Annealed and aged 500 hr at 1200°F	112.3	114.6	44.7	48.6	51	43
Annealed and aged 500 hr at 1300°F	112	113.4	44.5	47.6	49	41
Annealed and aged 500 hr at 1400°F	112.4	116	43.9	47	50	40

* Data from Oak Ridge National Laboratory.²⁵

of tests at 1200 and 1300°F are given in Table IV-13. Creep tests at stresses of 12,000 to 30,000 psi with specimens exposed to molten fluorides at 1100, 1200, and 1300°F have been initiated. The results of these tests are shown in Table IV-14.

The influence of compositional variations on the mechanical properties of INOR-8 has been studied⁴² by creep rupture tests at 1500°F and 10,000 psi. The results could not be explained

in simple terms of compositional variations since the principal factors of strength of the alloys are (1) solid solution elements, (2) carbide and noncarbide aging reactions, and (3) presence of MgC type carbide in the microstructure and grain size. The influence of chromium was found to be significant when 5 to 10 wt.% was added to 15 per cent molybdenum, balance nickel. The strengthening effect of iron was insignificant when amounts up to 10 wt.% were added to 15 wt.% molybdenum-7 wt.% chromium, balance nickel. The results are shown in Figs. 8 through 12.

A comparison of the creep-rupture properties of nickel in air and vacuum at temperatures from 1200 to 1500°F was made at the Naval Research Laboratory.⁴³ At very low stress, specimens tested in air at 1500°F appeared to undergo a marked strengthening accompanied by internal oxidation and virtual cessation of creep. The point on the deformation-time curves at which specimens tested in air became more

Table IV-12 ELASTIC PROPERTIES* OF INOR-8

INOR-8 heat	Temp., °F	Yield strength (0.2% offset), 1000 psi	Ultimate strength, 1000 psi	Ductility, %
Haynes SP-16	RT	45	106	58
Haynes SP-19	RT	45	114	39
Westinghouse	RT	52	117	50
Haynes SP-19	1000	27	90	19
Westinghouse	1000	36	100	43
Haynes SP-19	1100	29	93	50
Westinghouse	1100	38	103	37
Haynes SP-16	1200	25	67	44
Haynes SP-19	1200	27	82	36
Westinghouse	1200	38	83	16
Haynes SP-16	1300	24	58	37
Haynes SP-19	1300	28	70	24
Westinghouse	1300	38	70	18

* Data from Oak Ridge National Laboratory.²⁵

Table IV-13 RELAXATION DATA* FOR INOR-8

Temp., °F	Strain, psi	Initial stress, 1000 psi	Stress, 1000 psi, for constant elongation at		
			1 hr	10 hr	100 hr
1300	0.05	11	11.5	10	6
	0.1	21.5	21.5	16	5.5
	0.2	29.75	20.5	10.5	4.5
1200	0.05	12	12.5	12	10
	0.1	22.5	23	22	17

* Data from Oak Ridge National Laboratory.²⁵

Table IV-14 CREEP DATA* FOR INOR-8

Temp., °F	Stress, 1000 psi	Strain, %	Time, hr	Creep rate, %/hr
1300	30	15.33	110	Ruptured
	25	6.53	187	†
	20	9.56	882	Ruptured
	15	10.99	2894	Ruptured
1200	30	4.7	9†	Ruptured
	25	2.84	1195	2×10^{-3}
	20	2.70	1894	2×10^{-4}
	15	0.81	1863	2×10^{-4}
1100	12	0.97	1172	6×10^{-5}
	25	0.70	164	†
	15	0.58	68	†
	12	0.31	43	†

* Data from Oak Ridge National Laboratory.²⁵

† Test under way.

‡ Test to be repeated; low elongation at failure is not considered to be typical.

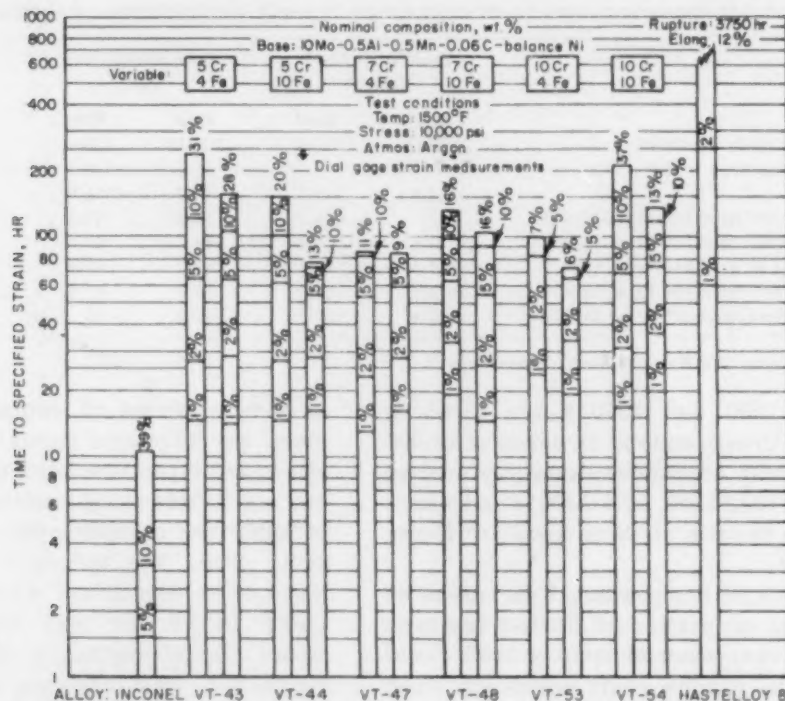


Figure 8—Bar graph of creep-rupture test results obtained at 1500°F, 10,000 psi, on nickel-base alloys with 10 wt.% molybdenum-0.5 wt.% aluminum-0.5 wt.% manganese-0.06 wt.% carbon and varying percentages of chromium and iron. Data from Oak Ridge National Laboratory.⁴²

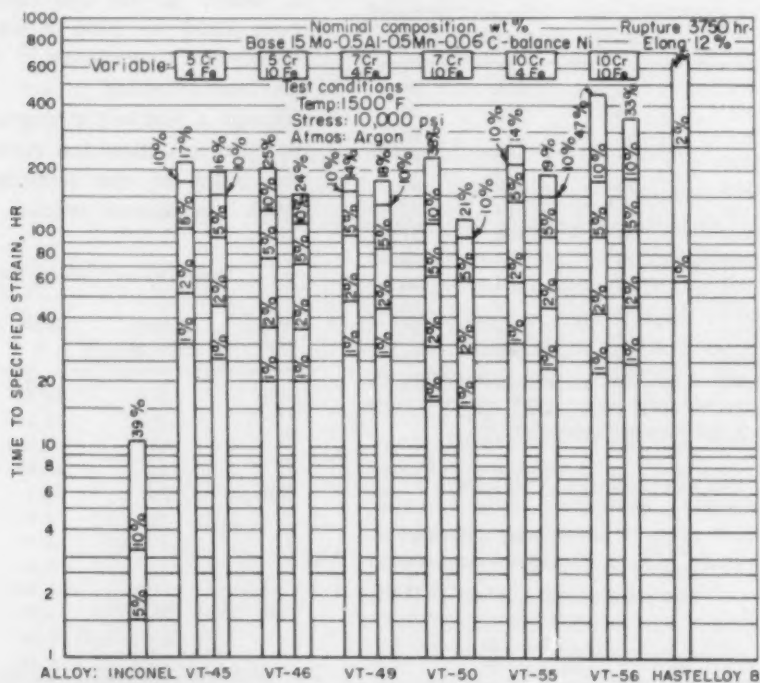


Figure 9—Bar graph of creep-rupture test results obtained at 1500°F, 10,000 psi, on nickel-base alloys with 15 wt.% molybdenum-0.5 wt.% aluminum-0.5 wt.% manganese-0.06 wt.% carbon and varying percentages of chromium and iron. Data from Oak Ridge National Laboratory.⁴²

creep resistant than those tested in vacuum was shifted toward longer times when the test temperature was reduced from 1500 to 1200°F.

A comparison of several nickel-base alloys, designated as INOR-1, 2, 3, 4, 5, and 6 (compositions given in Table IV-15) was made at Oak Ridge.⁴⁴ Stress-rupture properties were determined at 1350, 1500, and 1650°F on two individual heat-treatments of these materials.

(F. R. Shober)

Special Metallurgical Aspects of Cladding Materials

A dilatometric investigation of Zircaloy-2 by Bettis⁴⁵ has disclosed that the alpha to alpha-plus-beta transformation starts on heating between 815 and 830°C and ends on cooling between

785 and 770°C. The alpha-plus-beta to beta transformation ends on heating between 975 and 995°C and starts on cooling between 960 and 930°C. These data are in excellent agreement with established phase relations for zirconium-iron and zirconium-oxygen alloys. The zirconium-iron eutectoid is near 810°C, and a zirconium-0.2 wt.% oxygen alloy will finish transforming to beta at about 950°C. It is a generally accepted rule-of-thumb that the transformation range of zirconium is expanded by alloying elements to the temperature limits of the various binary systems involved.

A metallographic examination of zirconium ingots^{46a} has resulted in positive identification of one source of stringers in zirconium. The structures presented show that voids may be left in zirconium-alloy ingots melted under argon and that the fabrication of ingots containing such argon pockets results in "void type" stringers in the product. Other sources of stringers remain to be identified.

A minimum cooling rate of 50°C per minute is necessary to produce the required fineness of particle dispersion for maximum corrosion

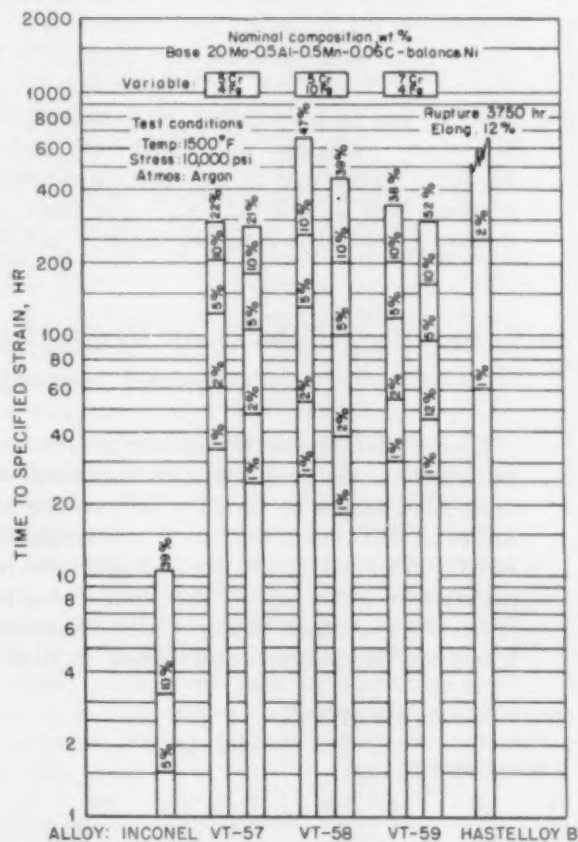


Figure 10—Bar graph of creep-rupture test results obtained at 1500°F, 10,000 psi, on nickel-base alloys with 20 wt.% molybdenum-0.5 wt.% aluminum-0.5 wt.% manganese-0.06 wt.% carbon and varying percentages of chromium and iron. Data from Oak Ridge National Laboratory.⁴²

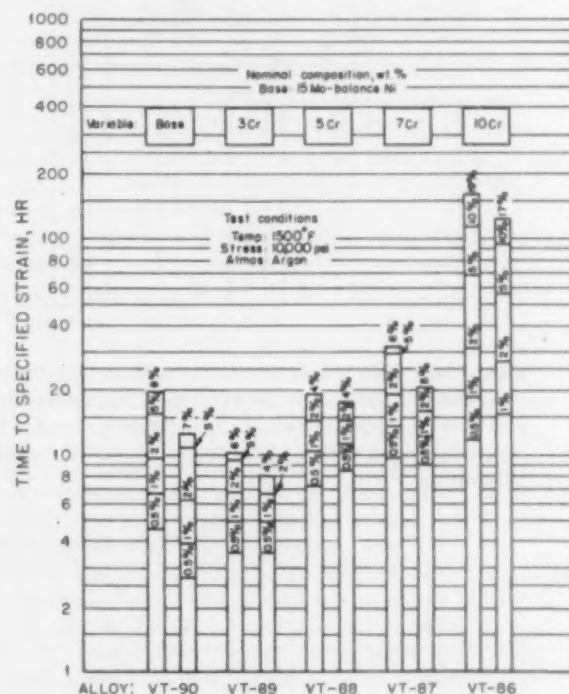


Figure 11—Bar graph of creep-rupture test results obtained at 1500°F, 10,000 psi, on nickel-base alloys with 15 wt.% molybdenum and varying percentages of chromium. Data from Oak Ridge National Laboratory.⁴²

resistance in Zircalloys.^{3,46b} Agglomeration of the precipitates in Zircalloys is undesirable for optimum corrosion resistance in water, and can be avoided by avoiding long treatments (over 1 hr) at temperatures above 700°C or by rapid cooling from above 870°C; the temperature range from 800 to 870°C is a critical one for Zircalloys and should be avoided as a heat-treatment range. Desirable dispersions (or supersaturated solutions) are best achieved by maintaining tempera-

tures below 800°C or by rapid cooling from temperatures above 870°C.

Atomics International is engaged in an investigation of the zirconium-rich corner of the zirconium-uranium-yttrium phase diagram.⁴⁷ No uranium-yttrium phase diagram was available, but on the basis of the data obtained, it was concluded that the solubility of yttrium in uranium is very small. Unpublished data on the zirconium-yttrium phase diagram, obtained from Denver Research Institute, show that this system contains no intermediate phases and consists of a simple eutectic at 1385°C and 56 wt.% yttrium. Terminal solubilities at 1385°C are about 2 wt.% for zirconium in yttrium and for yttrium in zirconium. The solubility of yttrium in zirconium at 865°C is about 0.6 wt.%. No room-temperature solubility is indicated. Yttrium was found to be about as insoluble in zirconium-uranium alloys as it is in pure zirconium; the solubility of yttrium in zirconium alloys containing up to 30 wt.% uranium is less than 5 wt.% yttrium at temperatures up to 1000°C, and is less than 1 wt.% below 550°C. A ternary eutectoid was found very close to the binary eutectoid in the zirconium-uranium system (600°C and 45 wt.% uranium). The ternary eutectoid is reported to occur at 550°C.

(W. Chubb)

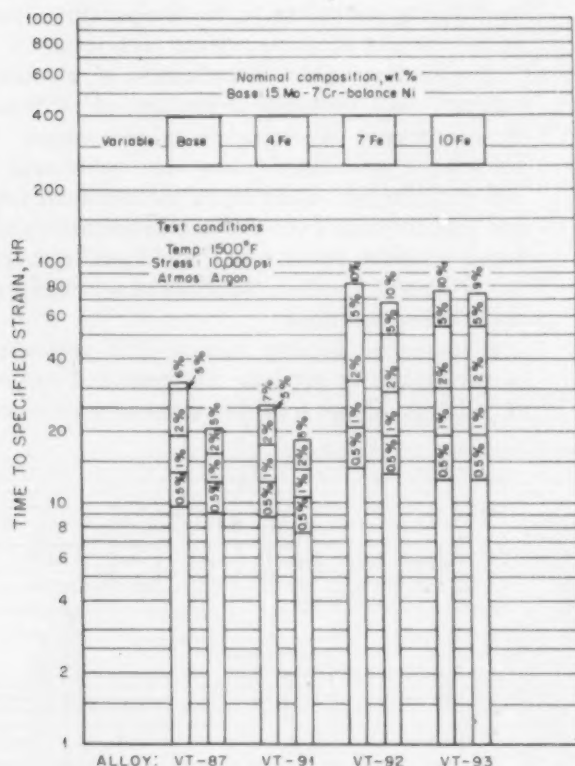


Figure 12—Bar graph of creep-rupture test results obtained at 1500°F, 10,000 psi, on nickel-base alloys with 15 wt.% molybdenum—7 wt.% chromium and varying percentages of iron. Data from Oak Ridge National Laboratory.⁴²

Irradiation Effects on Properties of Nonfuel Materials

Knolls⁴⁸ has studied the effects of neutron irradiation on the properties of mild steel when integrated fluxes up to 4.3×10^{20} nvt are used. ASTM A-201 steel was used and irradiations were carried out at a calculated specimen temperature of $300 \pm 100^\circ\text{F}$. The yield strength increased a maximum of 95 per cent. The ductility transition temperature increased an unknown

Table IV-15 COMPOSITION OF INOR ALLOYS*

Alloy	Composition, wt. %												
	C	Mn	Fe	S	Si	Cu	Ni	Cr	Al	Ti	W	Mo	Cb
INOR-1	0.01	0.47	0.28	0.002	0.47	0.02	78.44					20.29	
INOR-2	0.04	0.54	0.69	0.002	0.52	0.02	76.72	5.28				16.17	
INOR-3	0.01	0.53	0.18	0.001	0.57	0.02	79.86		1.08	1.62		16.11	
INOR-4	0.01	0.49	0.49	0.001	0.53	0.02	77.83		1.96	1.68		16.97	
INOR-5	0.10	0.94	1.66	0.002	0.68	0.02	78.59				2.69	13.15	2.15
INOR-6	0.02	0.57	0.59	0.003	0.62	0.02	74.01	5.30	1.15	1.69		16.01	

* Data from Oak Ridge National Laboratory.⁴⁴

amount, probably in excess of 400°F. It was found that recovery from radiation effects seemed to occur in the way predicted by the Arrhenius rate equation, as previously reported by Kunz and Holden⁴⁹

$$\tau = Ae^{-Q/RT}$$

where τ = recovery rate

A = a constant

Q = the activation energy

R = the gas constant

T = the absolute temperature

Harwell⁵⁰ has done similar work on En 2 steel, a low-carbon structural steel. Irradiations were carried out to a maximum integrated flux of 9.3×10^{19} nvt. The impact properties of the steel were investigated. It was found that the increase in transition temperature was approximately proportional to the cube root of dose. At maximum dose, the increase in transition temperature was 75°C. Also, it was found that specimens given maximum exposures absorbed only about half as much energy in ductile fracture as unirradiated specimens did.

In a study of the effects of irradiation on stress-relieved molybdenum and fully recrystallized tungsten by Makin and Gillies,⁵¹ wire specimens 0.040 in. in diameter were exposed at about 100°C to a total thermal flux of 5×10^{19} nvt. The fast-neutron integrated flux was estimated to be the same. The yield stress of molybdenum was increased by irradiation at all testing temperatures between 20 and 200°C, the increase being greater as the testing temperature was increased. A marked yield point was developed in the irradiated molybdenum after a 60-min 200°C anneal. The ductile-brittle transition temperature of the molybdenum was raised from -136 to -73°C by irradiation, whereas that of the tungsten was raised from 118°C to only 126°C. It was concluded that the magnitude of the increase in transition temperature was dependent on the initial transition temperature, being greater when the initial value was lower.

(D. C. Martin)

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Melting, Casting, Heat-treatment, and Hot Working

A study concerned with the factors that affect the species and quantities of nonmetallic impurities in vacuum-melted uranium has been concluded at Battelle.¹ One of the original aims of this study was to establish and measure the equilibrium pressures of gases with uranium. Thermodynamic calculations involving uranium at 2500°F indicated that the quantities of gases (other than hydrogen) present under equilibrium conditions were too small to be measured by the experimental techniques used. However, important information was obtained on the mode of reaction and the relative reactivity of each gas with the uranium-graphite system. Pressure-time data were gathered after measured quantities of N₂, H₂, CO, CO₂, and H₂O were admitted to a furnace in which molten unalloyed uranium in graphite crucibles was held at 2500°F. Reaction rates decreased rapidly as reaction-product films formed over the metal. The various films differed in their ability to inhibit further reactions. Those formed by O₂, CO, and CO₂ were highly effective in this regard. Films stemming from nitrogen and water were less effective. One of the most important observations was that the purity of the metal is sensitive to the type and amount of surface dross.

The effects of heat-treatment on the tensile and corrosion properties of Zircaloy-2 have been recently studied at Bettis.² The results, plotted in percentage changes because the data were obtained from longitudinal strip specimens from different ingots, are presented graphically in Figs. 13 through 15. Heat-treatments below 1750°F had little effect on the ultimate tensile strength or on the 0.2 per cent offset yield strength. Heat-treatments above 1750°F caused the yield strength to be higher and the ultimate strength lower than that of the as-received material. Both elongation and reduction in area decreased with increasing heat-treatment temperature, an increase in rate being noted at about 1650°F. Figure 15 shows how the corro-

sion rate by 750°F steam is accelerated by heat-treatment.

Hanford workers have been engaged in a comparison of methods of fabricating zirconium and Zircaloy-2 tubing.³ They studied welding and extrusion of various sizes of the tubing having various levels of cold work and concluded that: (1) a satisfactory method of fabrication exists for most of the sizes investigated (1.5 × 0.060 in., 1.5 × 0.035 in., 0.625 × 0.049 in., and 0.5 × 0.035 in.), (2) thin-wall large-diameter tubing (1.5 × 0.020 in.) is not amenable to fabrication, (3) with adequate precautions, corrosion during fabrication is not a problem, (4) mechanical properties which fulfill reactor requirements can be obtained in a variety of tubing sizes, and (5) yields are low, but can be improved with further development.

Sizable quantities of high-quality aluminum-25 wt.% uranium-alloy shot were prepared by the spinning-disk method by National Lead.⁴ This material is of interest because, during normal casting operations, the high uranium-containing component UAl₃ segregates by gravity. Nuclear Metals is hot compacting this material at 850°F at 65 tsi and will evaluate its qualities.⁵

The development of remote casting procedures for the aluminum-1.65 wt.% plutonium alloy has been reported at Hanford.⁶ Both static and vacuum-injection methods have been studied. In the evaluation of methods when AlSi is used, vacuum injection appears the most promising, and rods 3/4 in. in diameter and 8 ft 10 in. long have been cast in stainless-steel molds preheated to between 400 and 500°F.

(E. L. Foster)

Cladding

Roll Cladding

A limited number of Zircaloy-clad compartmented uranium fuel plates were rolled in the alpha, beta, and gamma temperature ranges in an attempt to roll clad uranium directly with Zircaloy.⁷ Although some of the plates fabricated

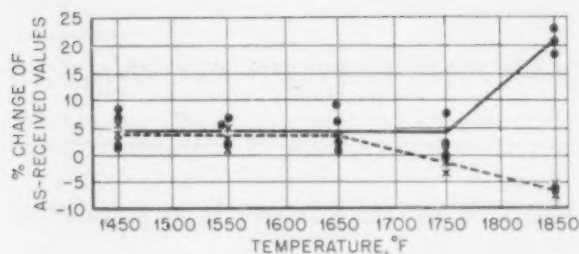


Figure 13—Percentage change of average 0.2% offset yield and ultimate tensile strength versus temperature of heat-treatment of Zircaloy-2 when compared with as-received values (samples held at temperature for 8 hr). —, 0.2% offset yield strength. ---, ultimate tensile strength. ●, Ingot No. 1. x, Ingot No. 2. Data from Bettis.²

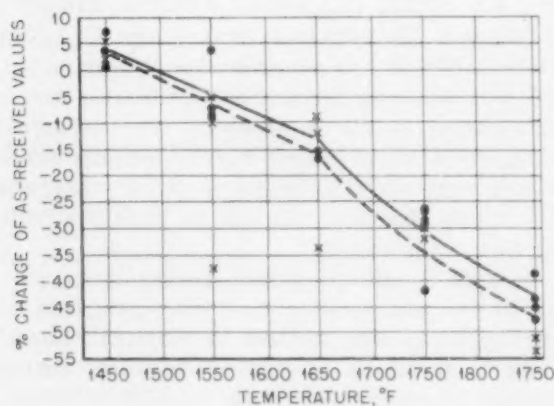


Figure 14—Percentage change of average elongation and reduction of area versus temperature of heat-treatment of Zircaloy-2 when compared with as-received values (samples held at temperature for 8 hr). —, elongation. ---, reduction in area. ●, Ingot No. 1. x, Ingot No. 2. Data from Bettis.²

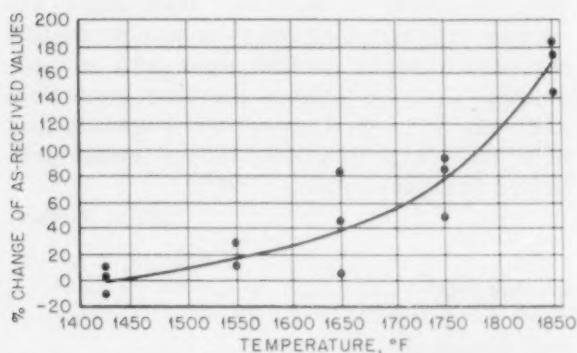


Figure 15—Percentage change of average weight gain versus temperature of heat-treatment of Zircaloy-2 when compared with as-received values (samples furnace-cooled after 8 hr at temperature; samples corrosion-tested 14 days in 750°F, 1500-psi steam). Data from Bettis.²

by a duplex roll-bonding process appeared to show promise, the plates exhibited intercompartmental leaks across the compartment bonds during corrosion testing. Roll cladding in the alpha range produced a good fuel-to-cladding bond and a poor cladding-to-cladding bond. Beta roll bonding resulted in poor bonding and poor dimensional control of the finished fuel elements. Increasing the roll-bonding temperature to the gamma range produced good bonds; however, dimensional control of the fabricated fuel plates was very poor. None of the roll-bonding temperature ranges gave both satisfactory bonds and satisfactory dimensional control.

The proposed Bettis⁸ reference process for the roll cladding of PWR Seed 2 elements includes the use of a total 6-to-1 reduction of a pieced composite sandwich. All of the Ti-Namel and Zircaloy components are prepared from piece components from cold-rolled and pickled stock. A savings in material of 30 to 60 per cent in Zircaloy-2 has been achieved through the use of the pieced Zircaloy components prepared from the as-cold-rolled and pickled stock.

Yankee Atomic Electric Company⁹ is considering a type 304 stainless-steel-clad 8 wt.% B₄C dispersion in silver as an alternative control rod. The element is to be fabricated by roll cladding at a temperature of 50°F below the melting point of the silver; consequently, it was necessary to roll clad the stainless steel to itself at 1650°F or lower and still obtain a stainless-to-stainless bond and a stainless-to-cermet bond. It was found possible to roll bond the stainless to itself at 1400 and 1650°F. The latter temperature was found to be preferable since it is sufficiently high to avoid carbide precipitation and to induce recrystallization across the bond interface. Preliminary results obtained by varying the roll-cladding schedule indicate that the best product is formed by starting with light initial passes and continually increasing the reductions until heavy reductions per pass are made. (E. S. Hodge)

Pressure Bonding

Battelle has clad nickel-plated tubular uranium slugs with aluminum by gas-pressure bonding.^{10,11} Optimum bonds were obtained in 1 hr at 950°F with a pressure of 10,000 psi. Lower pressures and a decrease in time produced weaker bonds. The strength of the bonds increased significantly with the formation of a

uranium-rich second phase at the interface. Specimens bonded for 5 min at 950°F exhibited a single-phase uniform-diffusion interface but poor bond strength. The width of the interface was not increased during a 1 hr-bonding cycle; however, the second phase was formed, and the bond strength was appreciably improved.

Pressure alters the thickness of the diffusion interface and affects the bond strength in various metallic systems. Studies at Sylvania have shown that, in the aluminum-nickel system, diffusion occurs by formation and growth of beta and gamma layers.¹² Pressure retards the growth of the gamma phase, and the chemical diffusion coefficient is reduced up to 27 per cent as the pressure is increased from atmospheric to 5 tsi. In contrast, other studies have shown that an increase in pressure promoted growth in the aluminum-uranium system.^{13,14} This suggests why the strength of the bond produced in the aluminum-clad-nickel-plated uranium slugs was improved by the use of higher temperatures and gas pressures.

Gas-pressure bonding is being utilized at Battelle for the preparation of Zircaloy-clad-flat-plate compartmented uranium dioxide fuel elements.^{10,11,15} In the current state of development, the specimens are pressure bonded in an autoclave at 1500°F for 4 hr with a helium-gas pressure of 10,000 psi. After the specimens are bonded they are heat-treated in a salt bath for 5 min at 1850°F and quenched. The postbonding heat-treatment completely transforms the Zircaloy into the beta phase. This promotes grain growth across the interface and generally improves the bond.

In these bonding studies, the surface preparation of the Zircaloy has a significant influence on the bond quality. Studies are being conducted with the objective of isolating the effects of conditions such as cold work, surface roughness, and the use of interface layers of carbon, chromium, iron, niobium, tin, and titanium.^{10,11,15} These tests have shown that surface roughness appears to exert the strongest influence on bond quality. A specimen with a 50 μ in. (rms) finish possessed partial grain growth across the interface. Specimens with coarser finishes exhibited complete grain growth.

A study is being conducted with the purpose of relating a basic property of a metal or alloy to its behavior during pressure bonding.^{10,11,15} It is assumed that the creep characteristic of the material is involved during the initial phase

of pressure bonding, when the components are forced into intimate contact and surface phenomena become active. A correlation will permit the prediction of pressure-bonding parameters of time, temperature, and pressure for any material whose creep properties are known.

(S. J. Paprocki)

Coextrusion

Two methods of coextruding Zircaloy-clad-uranium fuel elements from composite billets were developed by Nuclear Metals.^{5,16} Twelve elements were fabricated by the first method from four composite billets. The Zircaloy and uranium components of these billets were evacuated and welded closed before the assembly was canned in copper. This prevented air from contacting the surfaces that were to be bonded. Nine elements were fabricated by the second method from three billet assemblies. These billets used disks of uranium at the front and rear of the copper-canned and evacuated assembly to act as "getters" during preheating before extrusion. The results indicate that both methods provide satisfactory bonds and are equally good and economically competitive.

Nuclear Metals¹⁶ is conducting a program to develop methods to coextrude uranium tubes clad with Zircaloy-2 and having integrally extruded Zircaloy-2 end seals. The preparation procedures for the billet components and the selection of a billet design are of prime importance. Initial efforts have been devoted to obtaining reliable bonds in composite elements coextruded at relatively low reductions. Tests conducted on the bonds of the first series of specimens, extrusion bonded at 20:1, 15:1, and 10:1 reductions, showed the following: All Zircaloy-to-Zircaloy interfaces in elements produced at 20:1 and 15:1 showed good bonds, whereas some lack of bonding was indicated in the rod fabricated at a 10:1 reduction. All Zircaloy-uranium interfaces showed evidence of questionable or poor bonds.

The coextrusion of beryllium-clad uranium and of aluminum and aluminum-4 wt.% magnesium-clad uranium rods by the multitemperature extrusion technique has been established as being reasonably feasible.^{5,16} Cracking in the beryllium cladding has not been completely controlled. The experiments are described in somewhat more detail on pp. 14 through 17. It is tentatively assumed that furnace cooling is bene-

ficial because it allows slow relief of thermal stress and that water quenching is beneficial because it restricts the temperature rise in the uranium. (C. B. Boyer)

Welding and Brazing

In experimenting with several joining methods for uranium, workers¹⁷ at Los Alamos found that welding by the inert-gas-shielded tungsten-arc process is a successful technique if clean joint surfaces are obtained initially; also, good gas shielding must be maintained during and after welding. Optimum results were obtained with the following conditions.

Shielding gas: 90 vol.% helium - 10 vol.% argon.
Current type: d-c, straight polarity or a-c with superimposed high frequency for thin gauges.

Travel speed: 18.23 in./min.

Arc gap: 0.050 in.

Gas flow: torch, 60 cu ft/hr.

Gas flow: auxiliary, 60 cu ft/hr.

Current equations: (For sections 0.060-in. thick)

$$I = 1.5T + T/5$$

(For sections <0.060-in. thick)

$$I = 1.3T$$

where I is current in amp and T is thickness in mils of penetration desired. (For full penetra-

Table V-1 PHYSICAL PROPERTIES OF WELDED, AS-CAST, ROLLED, AND ANNEALED URANIUM*

Uranium	Tensile strength, 1000 psi	Yield strength,† 1000 psi	Elongation in 2 in., %
Welded	87	43	6-7
As-cast	52	21	8
HR-74% WR-86%			
As-rolled	205	105	4
Salt annealed (600°C)	145	48	7
Vacuum annealed (600°C)	140	69	14

*Data from Los Alamos.¹⁷

†Yield strength at 0.2 per cent offset.

tion, T equals the section.) Tensile properties of the welds are compared with those of uranium metal in various forms in Table V-1.

Ultrasonic soldering was demonstrated as a reliable method for joining uranium to itself and to other metals.¹⁸ Table V-2 lists pertinent information on the solders that were outstanding among a large group of compositions tested. Several tests made over a range of temperatures indicated that soldered joints were useful at temperatures between +80 and -80°C. Some loss in strength was generally noted above 40°C.

Table V-2 STRENGTH OF URANIUM-TO-URANIUM SOLDERED JOINTS*†

Solder	Melting point, °C	Flow point, °C	Composition, wt. %	Breaking load, lb	Shear strength, psi
A-9	78.9	78.9	57 Bi, 17 Sn, 26 In	647	1625
A-14	115.6	126.7	50 Sn, 50 In	325	793
A-18	177	177	67.7 Sn, 37.3 Cd	505	1137
B-1	156.4		100 In	252	700

*Lap joint: 1/2 by 3/4-in.; 40-mil sheet; soft solders tested at room temperature (average of five samples).

†Data from Los Alamos.¹⁸

Additions of approximately 0.2 wt.% niobium, chromium, tungsten, titanium, vanadium, and molybdenum to uranium were tested to determine their effects on weldability of the resulting alloys. Vanadium and chromium additions resulted in brittle welds. The remaining additives did not affect weldability adversely, but there was no indication that they improved it.

The University of California has been making tests of joints between beryllium and itself, stainless steel, and titanium. Aluminum filler material in beryllium-to-beryllium joints produced joint shear strengths of 25,000 psi. Pure silver gave the same strength in beryllium-to-stainless steel joints. Aluminum and copper-silver eutectic produced much weaker joints between these materials. Beryllium-to-titanium joints made with aluminum and silver appear to be satisfactory. Unclassified literature on the joining of beryllium was compiled by Oak Ridge.¹⁹

Resistance-bonding development at Knolls and Sciaky is continuing. A 600-kva machine is being built for welding full-scale fuel elements, consisting of Zircaloy cover plates and a zirconium-uranium core. A diffusion heat-treatment is required after resistance welding to obtain complete bonding. (R. E. Monroe)

Nondestructive Testing

Many applications and new developments in ultrasonic techniques for the inspection of materials and finished elements are reported. New equipment developed at the Sperry Corporation for Knolls will evaluate bonds ten times faster (600 interrogations/sec) than will present equipment. Ten-megacycle crystals will be used since this frequency appears to be optimum for minimizing the interference effects previously encountered with 5-Mc crystals. Recent experience has indicated that ultrasonic bond testing should be augmented by other techniques. Consequently, new methods of evaluating bond quality are being investigated, including ultrasonic Lamb-wave inspection, eddy-current techniques, infrared radiometric scanning, and electrical-potential resistance measurements.

An ultrasonic test for ingots has been developed by Bettis. The method employs a shear-wave crystal at 2.25 Mc in a specially built holder. It can detect a drilled hole $\frac{3}{64}$ -in. in diameter and $\frac{3}{32}$ in. long at $\frac{1}{4}$ in. beneath the surface.

At Du Pont,²⁰ an eddy-current instrument has been developed that will measure the thickness of copper plating on uranium with an accuracy of ± 5 per cent over a range from 0.004 to 0.012 in. The instrument is linear over this range and has good stability. It has also been used on copper or aluminum sheet up to 0.035 in. It is expected that it may be used on other materials by proper choice of frequency and sensitivity.

A radiation gauge is used at Atomic International²¹ to detect variations in UO_2 content in the stainless-steel matrix of the OMRE fuel-plate cores. A Ce^{144} source is used with a scintillation detector, digital counter, and auxiliary instrumentation. The equipment will easily detect 2 per cent relative variation of UO_2 concentration. Absolute calibration has not been obtained. Spot sampling is proposed for practical inspection since reading periods are of the order of 1 min per location.

Bettis has completed a water displacement unit for measuring metal volume and channel volume. It will measure volume with an accuracy of ± 0.1 per cent and reproducibility within less than 0.01 per cent. A new feature of the unit is an accurate placement control to allow the measurement of a selected portion of the length of a subassembly or cluster to within 0.010 in.

(C. V. Weaver)

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